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INTEGRATED RETORTING-COMBUSTION SYSTEM USING FLUIDIZED BED TECHNOLOGY

FINAL REPORT
BY:
POLYMATH ENERGY CONSULTANTS LTD.
AND
DEPT. OF CHEMICAL ENGINEERING, UNIVERSITY OF BRITISH COLUMBIA

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1.0 EXECUTIVE SUMMARY

A successful 48 hour test run was carried out on the UBC fluidized bed, retorting co-combustion pilot plant - with pyrolysis of New Brunswick Oil Shale in the retort, and spent shale and Minto coal fuelling the combustor. Results of the test are presented. The primary purpose of the test was to determine the suitability of the apparatus, and thus no optimization or investigative program as indicated by the results was followed.

The oil yield from the shale was 44% of the Fischer Assay evaluation. This is lower than previous studies have reported, and is probably due to cracking or re-deposition of oil on certain mineral species within spent shale that had a long residence time in the fluidized bed reactor.

Sulphur capture was 45% in the combustor. This value was directly due to the small amounts of CaO in the spent shale fed to the combustor. The calculated Ca/S mol ratio was 0.52, and if this ratio was raised to unity or above the sulphur capture should increase.

For a shale feed to the retort of 3.78 kg/h, and a coal feed to the combustor of 2.37 kg/h, the unit heat loss at 29,304 kJ/h amounted to about 41% of the heat input. The discharged flue gas contained about 39% of the heat input. For larger units than this test apparatus this amount should be higher, and about half of this lost heat could be recoverable as useful energy.

1.0 RAPPORT ADMINISTRATIF

Un essai de fonctionnement d'une durée de 48 heures fut mené avec succès à l'UBC sur un système-pilote composé d'un four de fluidisation à pyrogénation par co-combustion - avec de la pyralyse du Schiste Bitumineux Nouveau Brunswick dans la cornue, et du schiste décomposé et de l'houille Minto alimentant la chambre de combustion. Les résultats de l'essai sont ici présentés. Le propos premier de l'essai ayant été de déterminer les capacités de l'appareil, aucune épreuve d'optimisation ou autre programme de recherche furent, d'après les résultats, entrepris.

L'huile débité par le schiste atteignit une valeur de 44% suivant la méthode d'évaluation dite Analyse de Fischer. Cette valeur est inférieure à celle rapportée par des études précédentes, et résulte probablement du craguage ou la re-déposition des huiles dans certaines espèces minérales contenues dans le schiste décomposé lesquelles ont un temps de séjour très long dans le réacteur du four de fluidisation.

La capture en soufre dans la chambre de combustion représenta 45%. Cette valeur étant le résultat direct des petites quantités de CaO contenues dans le schiste décomposé qui alimente la chambre de combustion. Le ratio Ca/S mol fut de 0,52, et au cas où ce ratio était augmenté à des valeurs égales ou supérieures à l'unité la capture en soufre devrait s'accroître.

Pour une alimentation en schiste de la cornure de 3,78 Kg/h et une alimentation en houille de la chambre de combustion de 2,37 Kg/h, la perte en chaleur de l'appareil à 29,304 Kj/h se ramena à environ 41% de la chaleur fournie. Le gaz évacué de la chambre contenait environ 39% de la chaleur fournie. Pour des systèmes plus larges que cet appareil d'essai ce montant devrait être plus important, et environ la moitié de la chaleur perdue pourrait être récupérée en energie utilisable.

2.0 INTRODUCTION

The pyrolysis of New Brunswick Oil Shale in a retorting unit has been examined in several previous studies, and information on oil yields obtained. However, the co-processing of the oil shale with a high sulphur New Brunswick coal, where the coal provides a source of heat for retorting with a possible surplus of heat for power generation, has many attractive features. If this objective could be attained using fluidized bed technology for combustion in order to keep sulphur and other noxious emissions at low levels, then the system would also fulfil requirements for thermal power plants now in force.

CANMET initiated and requested proposals for the above concept. and decided in July, 1986, as a consequence of the responses in the form of proposals, to have single experiments performed by certain contractors. Each experiment was to be of a comprehensive nature that would determine the capability of the various test apparatus and of the contractor's performance. Polymath Energy Consultants Ltd. was awarded a contract in November, 1986 to undertake one experiment, in association with the Department of Chemical Engineering, University of British Columbia. A basic integrated pyrolysis-combustion unit was available at the University.

A brief description of the unit as it was initially available is of interest. The unit was a two stage direct heating system with an upper pyrolysis stage and a lower combustion stage. Spent carbonaceous solids from the upper pyrolysis chamber could be fed to the lower refractory-lined combustor to add to other fuel or to fully provide the heat for the pyrolysis process. The lower stage could be operated under neutral or slightly reducing conditions. Steam was used to temper the combustion process and an interstage cooler provided additional flexibility in temperature control. The combustor was originally operated in the spouted bed mode but could equally well be used in the fluidized bed mode. As initially suggested by Polymath this unit could be used in a direct heating combustor-pyrolysis arrangement.

The contract given to Polymath specified the modification of the above equipment to provide an indirect heating of recycled retort gas for pyrolysis, and it required that a steady-state test of sufficient duration for the experiment to show the suitability of the apparatus. This report presents the apparatus final arrangement and description, test conditions, performance, results and conclusions obtained in the fulfillment of the contract.

3.0 TEST OBJECTIVES

The general objective of the test work was to perform a steady-state experiment of sufficient duration to prove out the test equipment, and to provide data to evaluate the integrated system using fluidized bed technology. The following particular objectives were also specified in the contract:

A. An integrated pyrolysis-combustion system was to be used.

B. New Brunswick Oil Shale was to be used for pyrolysis.

C. New Brunswick Minto Coal was to be used for combustion.

D. The steady-state portion of the experiment was to have a duration of at least 48 hours.

E. The fluids from the retort were to be separated from the solids and cooled to produce a retort gas stream and a retort liquid stream.

F. A portion of the retort gas stream was to be reheated by heat exchange by the hot combustor flue gas stream, and recycled through the retort to provide the heat for pyrolysis as well as the flow to fluidize the shale material. By using this indirect arrangement the flue gas stream from the combustor coal does not enter into nor contaminate the retort gas stream.

4.0 PREPARATIONS AND MODIFICATIONS

This section briefly describes the procurement and analysis of the materials specified for the test, and the modifications required to adapt the UBC integrated pyrolysis-combustor unit to meet the specified requirements. A detailed description of the test facilities as finally set up for the experimental program, with a flow diagram, is given in Section 5., following.

4.1 Procurement and Analysis

New Brunswick Oil Shale and Minto Coal were ordered in mid-December from the Research and Productivity Council of New Brunswick. They had an immediately available supply on hand, and very generously accomodated us with the material, saving considerable delay that could have been experienced if the shale material had to be collected.

One thousand kilograms (1000 kg) each of the shale and coal were ordered and supplied. The material was shipped in barrels via CP Rail on January 9, 1987. It was delivered at the University of British Columbia on February 24, 1987.

Crushing and sampling was commenced immediately. With this type of material, and in the quantities concerned, the process of crushing and screening is a relatively slow and manually controlled process using the equipment available in the UBC laboratories. Nevertheless the materials were reduced to the required size, and composite samples of each were taken for analysis, before the crushed material was transported to the experiment site and stored.

The following analyses were performed:

Coal	Shale
Proximate	Proximate
Ultimate	Ultimate
Heating value	Fischer assay
Mineral matter analysis (SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, etc. as %.)	Mineral matter analysis
Particle size analysis	Particle size analysis

The above analyses, with the exception of the Fischer assay, were performed by Chemex Labs Ltd., of North Vancouver, BC, and their attested results are given in Appendix 1a.

The oil shale Fischer assays were performed on three samples of varying sizes at the New Brunswick Research and Productivity Centre. The results obtained are given in Appendix 1b.

4.2 Suitability of Existing Equipment

The pyrolysis-combustor unit at the University of British Columbia was examined, and the arrangement compared to the conceptual flowsheet as it had been developed during the discussions leading to the contract. The unit had previously been sucessfully used in tests with New Brunswick shale, so its basic suitability was not in question.

Two major differences in the arrangement and attached facilities were obvious. The existing unit directly connected the combustor flue gas outlet to the retort inlet immediately above. The criteria for the experiment prescribed an indirect heating of the pyrolysis gas. This requirement was met by constructing a spool piece, sealed against through passage of the gas to separate the two reactors. A flue gas exit flange allowed the hot flue gas to exit the combustor side of the spool piece to pass through a new heat exchanger, before it was vented from the building.

The heat exchanger had the recycled retort gas arranged to pass through the other side, satisfying the indirect heat exchange requirement. A small positive displacement blower was deemed necessary to provide the pressure needed to pass the recycled retort gas through the heat exchanger and to provide the pressure necessary for fluidization in the retort.

The second major difference was the provision of a new, high temperature, heat conserving cyclone to clean the flue gas stream. A refractory-lined cyclone was designed and constructed to meet this requirement.

Several other minor differences were modified as needed:

- distributor plates in both the combustor and retort established fluidized bed modes.

- two demisters in parallel were deemed necessary for the retort gas stream, with one on line at a time. This was a precaution in case of demister blockage.

- an electrostatic precipitator provided a final clean-up of the retort gas stream.

- some changes in piping, connections, pressure, temperature and flow taps,and in support structure were needed.

It was decided, at this initial stage, to retain the basic vertical, interconnected compact arrangement, even though the through flow of gas had been changed. This decision later proved to be a source of difficulty and delay in establishing piping and other interconnections due to the spacial restrictions involved.

Before any modifications commenced the flowsheet and details of the reconstruction were reviewed by the Scientific Authority.

5.0 DESCRIPTION OF THE TEST APPARATUS

The test apparatus, and the flows through the system, are shown schematically in Figure 1. The apparatus comprises the following main components:

5.1. **Fluidized bed combustor** (Figure 2)

This is a refractory-lined cylindrical vessel, 150 mm i.d. x 1.2 m high. The combustor has a fuel feed hopper and rotary valve feeder which normally feeds about 5 kg/h of coal in the size range 0 - 6 mm.

A number of overflow pipes are supplied to control bed level. The combustor is equipped to burn propane for start-up and has a propane pilot burner, spark igniter and UV flame detector.

A perforated stainless steel plate at the bottom of the combustor serves to support the sand/fuel bed and to distribute the fluidizing air into the vessel. The combustor operates at atmospheric pressure or at a slight positive pressure, with superficial fluidizing velocities ranging from about 0.2 - 3.0 m/s. Temperatures can be taken as high as 1050°C.

Connections are supplied below the distributor plate for the supply of air, nitrogen, propane and steam (if needed). Connections are supplied above the distributor plate for the addition of solid fuels, the removal of bed samples, and the escape of flue gas via a central or side outlet. Connections are also available for thermocouples and for a small cooling coil in the upper combustor freeboard space.

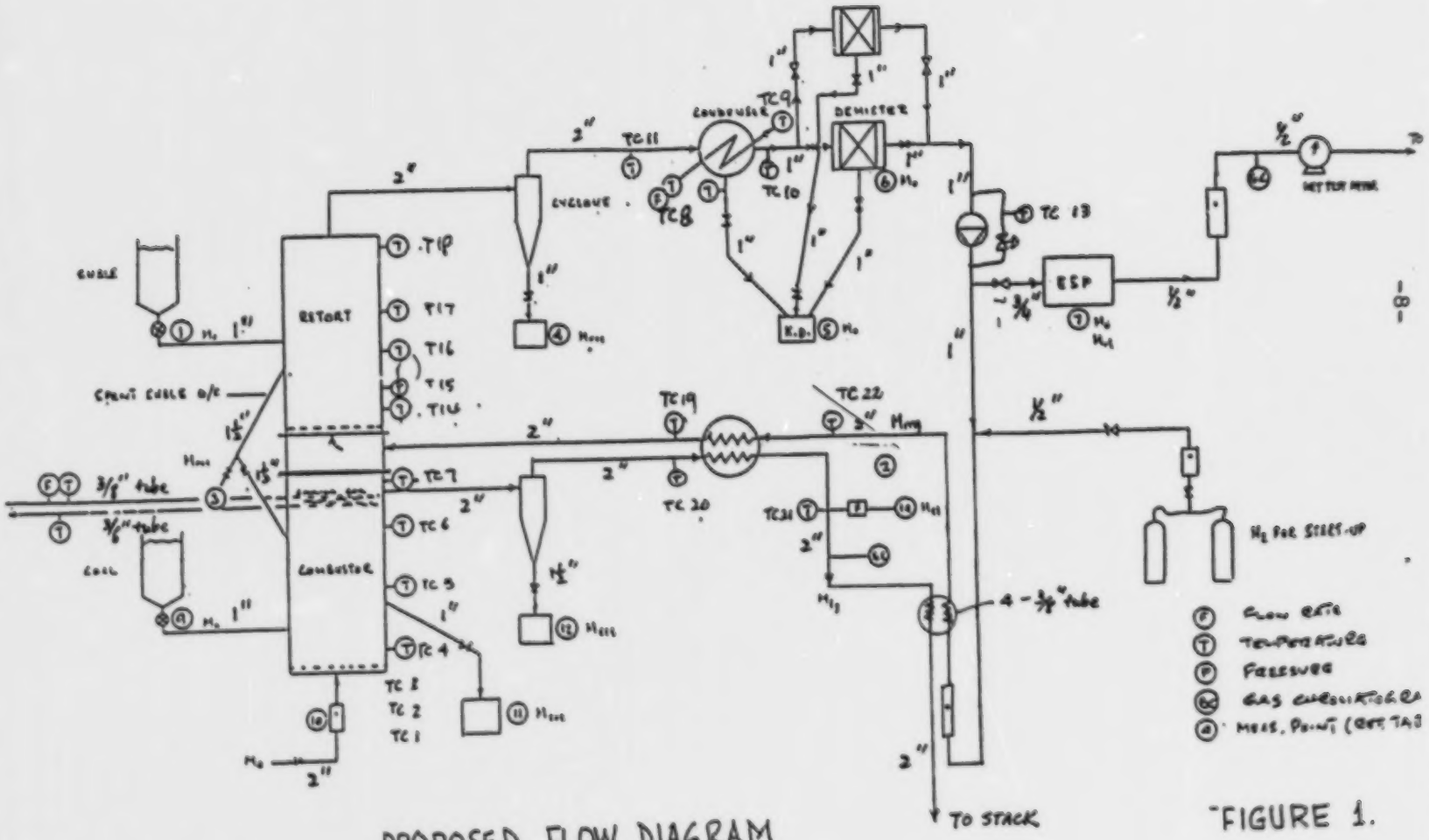
5.2 Fluidized bed retort (Figure 2)

This is a Type 316 stainless steel cylindrical vessel, 150 mm i.d. x 1.0 m high, that rests concentrically above the combustor. The retort has a shale feed hopper and rotary valve feeder which can feed up to 12 kg/h of shale in the size range 0 - 6 mm.

A perforated stainless steel plate at the bottom of the retort supports the shale bed and distributes the fluidizing gas flow. The retort operates at atmospheric pressure or at a slight positive pressure, and can operate under reducing, neutral or oxidizing conditions.

Overflow pipes are supplied to control bed level and to extract spent shale from the retort. From the discharge point at 240 mm above the base the spent shale runs down an inclined tube into a receiver which can be removed from the line after closing a plug cock to seal the line.

The retort can be operated at temperatures between 500 and 700°C. Bed depth is sensed via a pressure probe and the bed discharge rate may be set to maintain constant level and steady-state conditions.



PROPOSED FLOW DIAGRAM

(Updated)

FIGURE 1.

PLEASE NOTE:

There was to have been a photograph on this page but since it was not required by the contract it has been deleted.

5.3 **Combustor cyclone** (Figure 3)

Dust laden flue gases leaving the combustor at 850 - 1000°C enter a refractory-lined high efficiency cyclone (100 mm i.d. x 530 mm high). The cyclone is designed to remove over 99% of particles leaving the combustor before the gases enter heat exchangers, and to retain as much heat in the gas as possible.

5.4 **Retort cyclone**

Hot dust laden gas and vapours from the retort pass through a stainless steel cyclone to separate solids before the vapours enter a water cooled condenser.

5.5 **Vapour condenser**

Hot gas and vapour leaving the retort cyclone enter a shell and tube heat exchanger, with 4 ft^2 tube surface area, where the gas is cooled to about 30°C. Condensed hydrocarbon liquid drains from the bottom of the heat exchanger into a receiver. Cooling is provided by city water. The cooled gas passes into parallel demisters (2) then into the suction side of the retort gas blower.

5.6 **Retort gas blower** (Figure 4)

Retorting gas is recirculated through the retort via a rotary lobe positive displacement type blower (Sutorbilt California Series F, Model 2MV, direct drive, 1750 RPM) The blower is equipped with a water-cooled by-pass loop, (installed for this experiment), by which the net blower discharge can be varied, while keeping the blower cool. The blower discharge gas passes via a rotameter into the gas/gas heat exchangers where it is reheated to retorting temperature by the hot flue gas from the fluidized bed combustor.

5.7 **Gas-to-gas heat exchangers**

Flue gases exiting the combustor cyclone enter two shell and tube heat exchangers in series. The flue gases flow on the shell side of the heat exchangers while a countercurrent flow of retorting gas flows on the tube side.

The first heat exchanger contains stainless steel U-tubes having 4 ft^2 of heat exchange surface area. The second heat exchanger contains multiple straight stainless steel tubes, having 3.5 ft^2 of heat exchange surface area. Retorting gas is heated from about 100°C to 500 - 550°C in the heat exchangers. Initial calculations indicated that between 5 to 7 ft^2 of heat exchange surface was needed. An early test showed that the 4 ft^2 exchanger was just insufficient, thus the second exchanger was designed and added.

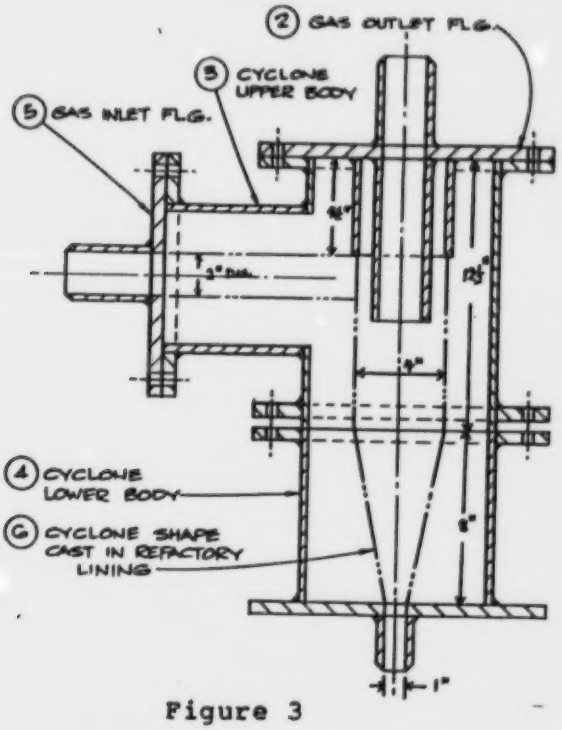


Figure 3

Detail of Refractory-lined Combustor Cyclone

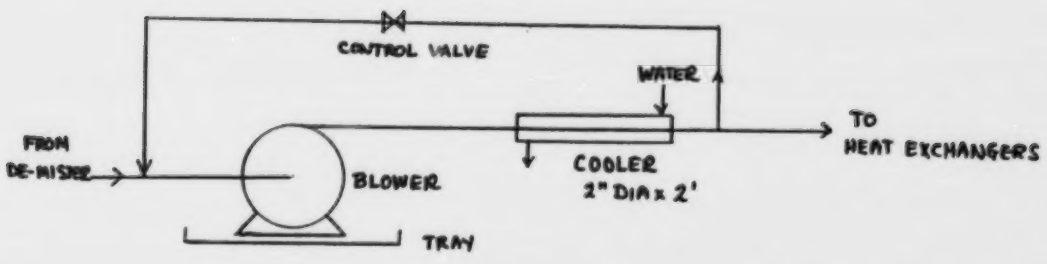


Figure 4

Blower Arrangement

(Note by-pass cooler and trays collecting kerogen)

5.8 **Electrostatic precipator** (Figure 5)

Excess quantities of retorting gas are vented from the retort loop through an electrostatic precipitator (ESP) and gas meter. The ESP removes fine particles prior to the gas exiting the system. As the gas flow involved is not large the ESP is a simple arrangement of four tubes each with a central wire.

5.9 **Miscellaneous**

Sizes for the main items of equipment are given as part of their description. Minor features, piping, fuel and shale feeders, etc., are generally dimensioned in the sketch of Figure 1. Construction sketches and photographs of the various items of equipment were made available to the Scientific Authority, and are on file.

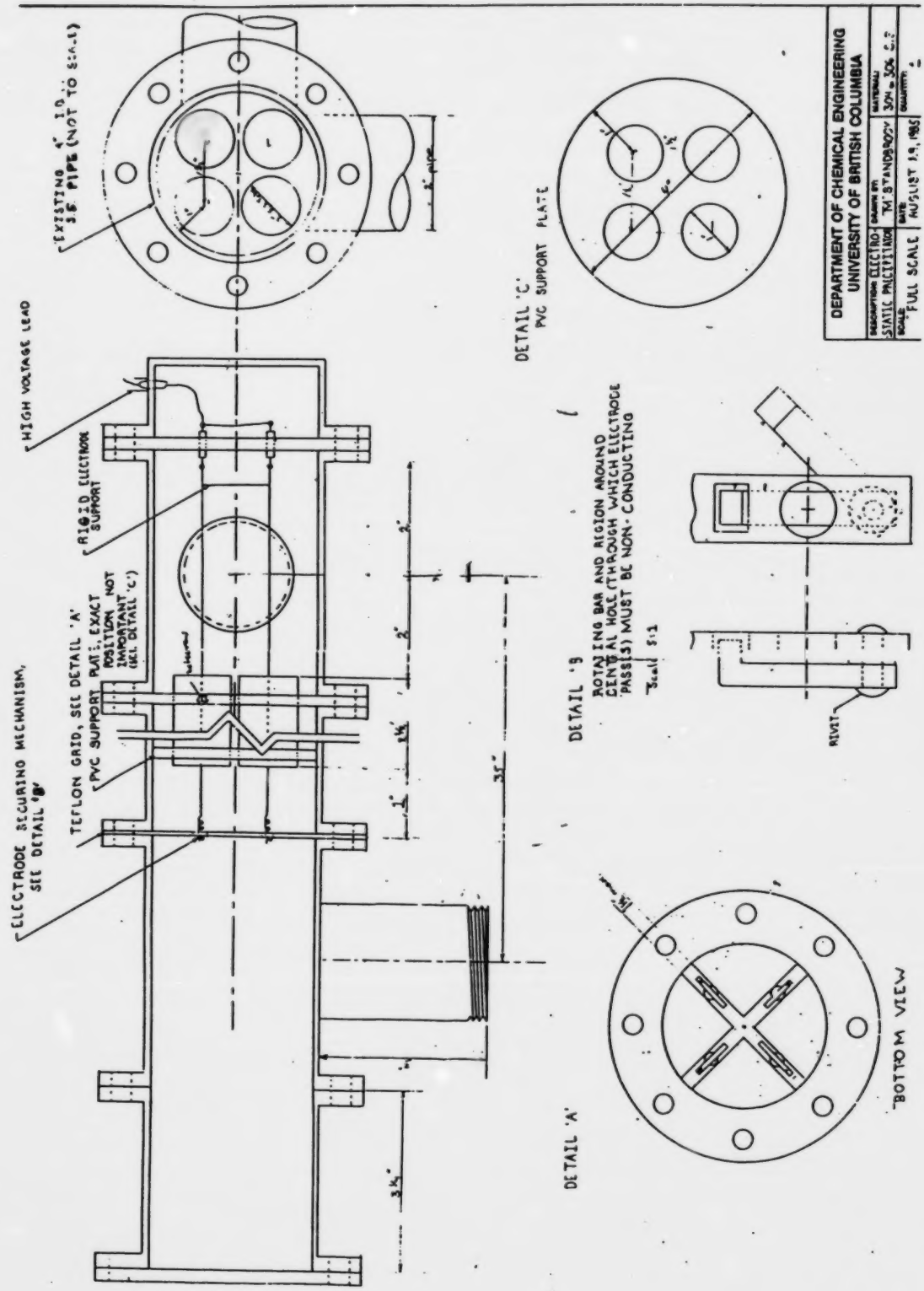

EXISTING 4" I.D. 16 PIPE (NOT TO SCALE)
HIGH VOLTAGE LEAD
RIGID ELECTRON SUPPORT
TEFLON GRID, SEE DETAIL 'A'
PVC SUPPORT PLATE, EXACT POSITION NOT IMPORTANT (SEE DETAIL 'C')
ELECTRODE SECURING MECHANISM, SEE DETAIL 'B'
DETAIL 'C'
PVC SUPPORT PLATE
DETAIL 'B'
ROTATING BAR AND REGION AROUND CENTRAL HOLE (THROUGH WHICH ELECTRODE PASS(S)) MUST BE NON-CONDUCTING
Scale 5:1
RIVET
DETAIL 'A'
BOTTOM VIEW
DEPARTMENT OF CHEMICAL ENGINEERING
UNIVERSITY OF BRITISH COLUMBIA
DESCRIPTION: ELECTROSTATIC PRECIPITATOR
DRAWN BY: M. STANDISH
MATERIAL: 304 - 304 S.S.
SCALE: FULL SCALE
DATE: AUGUST 19, 1985
DRAWING #: 2

FIG. 5 ELECTROSTATIC PRECIPITATOR

6.0 PERFORMANCE OF THE EXPERIMENT

A narrative description of the experiment progress is given in this section. Itemized procedures for start-up and operation, shut-down, and emergencies are given in Appendix 3, though they were followed during the experiment.

6.1 Introduction

A requirement of the contract was an experimental run duration of 48 hours. The 48 hour test-run was attempted twice. The first attempt had to be abandoned 20 hours after start-up because of solids hold-up in the retort, and the formation of a large sinter in the combustor. After shut down the unit was modified to correct these problems, which originated because combustion gases were back-feeding through the spent shale feed line preventing a proper spent shale feed. The unit was then readied for the second test attempt. This test took place on July 20 - 22, 1987, and was successfully carried through. Events during the two test runs are described below.

6.2 First Test Attempt

Following the completion of all initial modifications the pilot-plant test unit was checked and calibrated in a series of smaller runs. A final test of 12 hours duration proved all parts to be operating as expected, and it was decided to commence the 48 hour test run on July 15, 1987.

The pilot plant was started up on propane, then brought up to operating heat conditions on a Western Canadian (Forestburg) coal, before the Minto coal started feeding to the combustor. Light-up of the propane burner was at 0800 hours. The combustor and retort were at operating temperature by 2300 hours and had been solely on Minto coal for a sufficient period. At this time shale feed was commenced to the retort section.

The unit operated steadily for about 2 hours, but at about 0100 hours it was noted that the shale was not feeding properly to the retort. It was suspected that combustion gases were passing up the spent shale return leg into the retort, and in doing so were interfering with the flow of spent shale into the combustor.

After consideration, it was decided that a second valve should be installed in the spent shale return line, so as to function as a small lock hopper that could be manually operated, allowing spent shale to feed into the combustor without back flow of combustor gas. Accordingly the unit was shut-down at 0200 hours for about 20 minutes while the valve was installed.

When a re-start of the combustor was tried, it was found impossible to maintain a uniform bed temperature -,i.e. the bottom

of the bed was cold while the top was hot. It was concluded that a large amount of sinter (or clinker) had formed in the bed during the shut-down period, preventing it from fluidizing properly.

At this point the decision was taken to shut down and clean the pilot plant and to abandon this first test run. The combustor bottom and distributor plate were removed and a very large sinter mass was found to have bridged across the upper part of the combustion chamber. Later, the retort was also opened and was found to be blocked with solids. From this it was deduced that the spent shale had not been leaving the retort section in the expected manner, and that good fluidizing conditions were not being achieved in the retort. A number of changes were decided on and put into effect to remedy these problems. These changes were:

- a spent shale receiver was placed at the end of the spent shale overflow line. Spent shale that collected in this receiver was then manually blended with Minto coal and then fed to the combustion chamber via the coal feed system. In effect a manual lock hopper arrangement was temporised.

- a U-tube manometer was installed across the retort bed so that differential pressure could be noted and, more importantly, so that small pressure oscillations typical of fluidized bed operation could be observed.

- the blower suction lines were equipped with glass fibre filters to remove kerogen passing the demisters and entering the blower.

Following these changes it was decided to reschedule the test for July 20,1987.

6.3 **Second (Successful) Test Run**

The unit was started at 0800 hours on July 20, 1987 by firing it with propane, followed by using Forestburg coal to reach operating temperature . Then Minto coal feed commenced. By 1545 hours conditions were steady at 955^{o}C but it was proving difficult to fluidize the retort because the blower was overheating. The blower suction lines were changed to larger diameter tubing and the suction filters were removed. Blower performance was then adequate to fluidize the retort without overheating.

At 2145 hours the shale feed was started to the retort. Combustor temperature was 925^{o}C and the retort was at 460^{o}C. Both the combustor and the retort were very stable with only minor temperature fluctuations. The mixture of coal and spent shale going to the combustor burned evenly, and temperatures in the combustor ranged through only a 2 - 4^{o}C variation.

The test was considered to be at steady-state and commencing at 2200 hours.

It was noted that the blower was leaking oil from vent points. Trays were put beneath the blower to catch the oil, since it was obviously part of the oil yield from the shale, being condensed in the blower, or cooled in the by-pass.

From the start of the shale feed (2145 hours) until about 0615 on July 21, 1987, the pilot plant ran very smoothly and stably. No problems were encountered with the shale feed, the spent shale overflow, or the coal feed.

Minor problems that were noted, but ignored unless they became serious, were:

- a leak developed in a flange on the line where the hot recycle gas entered the retort. It was the result of a weld crack and could not be repaired without shutting down the unit. It was decided to continue operating the test program, even though some minor amount of hydrocarbons and recycle gas were being lost. In an attempt to reduce the effect of this loss the crack was partially sealed by application of a high temperature cement to the crack area. This improved the situation but did not completely solve it.

- nitrogen consumption was substantial as a result of the above leak.

- kerogen vapour carried past the demisters and into the blowers. On compression, the kerogen condensed and leaked from the blower vents. The leakage was collected in trays and added to the reported catch. Some kerogen also carried over into the recycle gas, as evidenced by coating of the rotameter tube, but was undoubtedly minor.

Despite the above small problems the unit operation was very stable. It was, in fact, so steady and predictable that it lulled the operators so that when a more serious problem arose it caught them unprepared and almost ended the test run.

At 0615 hours the coal feed line plugged, probably due to a sinter forming, or coal caking at the exit into the combustion chamber. The line was disconnected, back-flushed with combustion air, and prodded out to clear it. By the time this was completed the combustor temperature had dropped to 650°C, with the lower bed portion showing less than 500°C.

The unit was restarted by adding a small amount (about 0.25 kg) of Forestburg coal via the coal feed line just before it was reconnected. The volatile coal ignited easily when the fluidizing air was reestablished, and allowed the unit to be brought back under control. It was felt the small amount of Forestburg coal need not cause the test run results to be compromised, provided the remainder of the long test run was uneventful. The delay and rapid drop in temperature were more serious, as the unit was still recovering at 0800 hours , and the retort temperature was just at 500°C by that time, but the test was continued.

After that brief flurry of "excitement", the remainder of the test run was noteable for its lack of drama. Combustor and retort feed and control systems operated continuously and without major problems for the last 40 hours of the test program. A few minor running difficulties did occur, as with any experimental apparatus, but these were taken care of as the test proceeded. These minor problems did not interfere with the test program and included: a temporary loss of nitrogen supply due to a faulty level gauge on the liquid nitrogen tank; a fuel hang-up in the rotary feed valve, easily cured by a hammer.

The pilot plant was operated until 2200 hours on July 22, 1987, when the last samples were taken and the experiment duration was considered met, and the unit was shut down. Total running time was 62 hours, of which slightly more than 48 hours were spent with the shale and coal feeding simultaneously to the retort and combustor respectively. The problem experienced, and its recovery, were considered to be of small impact on the overall test run results as no other major delay or alteration of fuel was needed.

6.4 **Lessons learned from the Test Runs**

A number of valuable lessons were learned from the test run experiment. The first, and important lesson from the viewpoint of the Polymath/UBC team, was that the UBC pilot plant is fully capable of operating continuously over long runs, of 48 hours or more, and this justified the faith and claims made for the unit in the proposals.

Other practical lessons learned included:

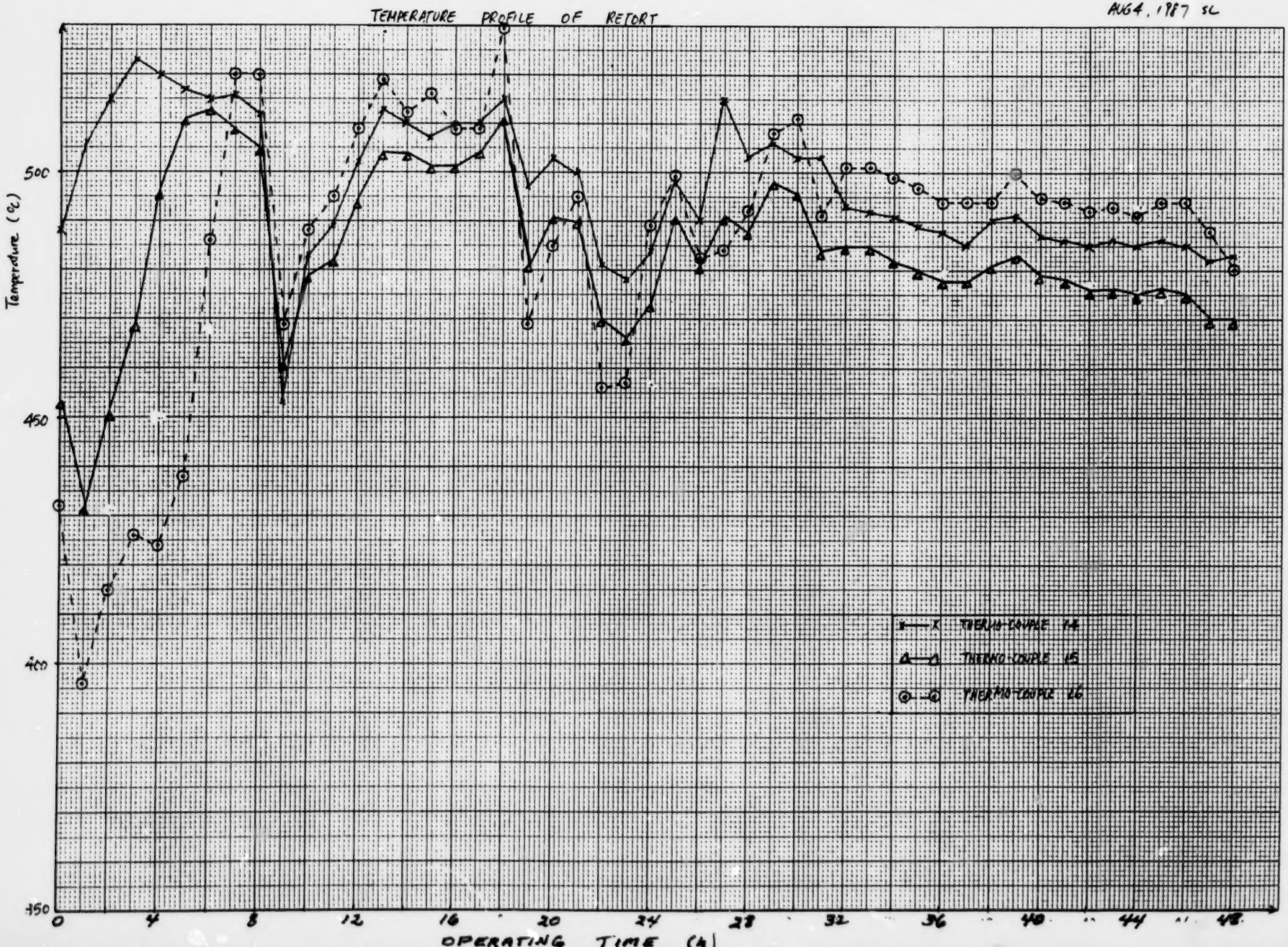
- effective spent shale discharge from the retort feeding into the combustor requires a lock hopper and a dedicated feeder. Alternatively, an "L" valve might be effective, although the coarse, granular nature of the material might cause it to hang-up in line sections without a steep gradient.

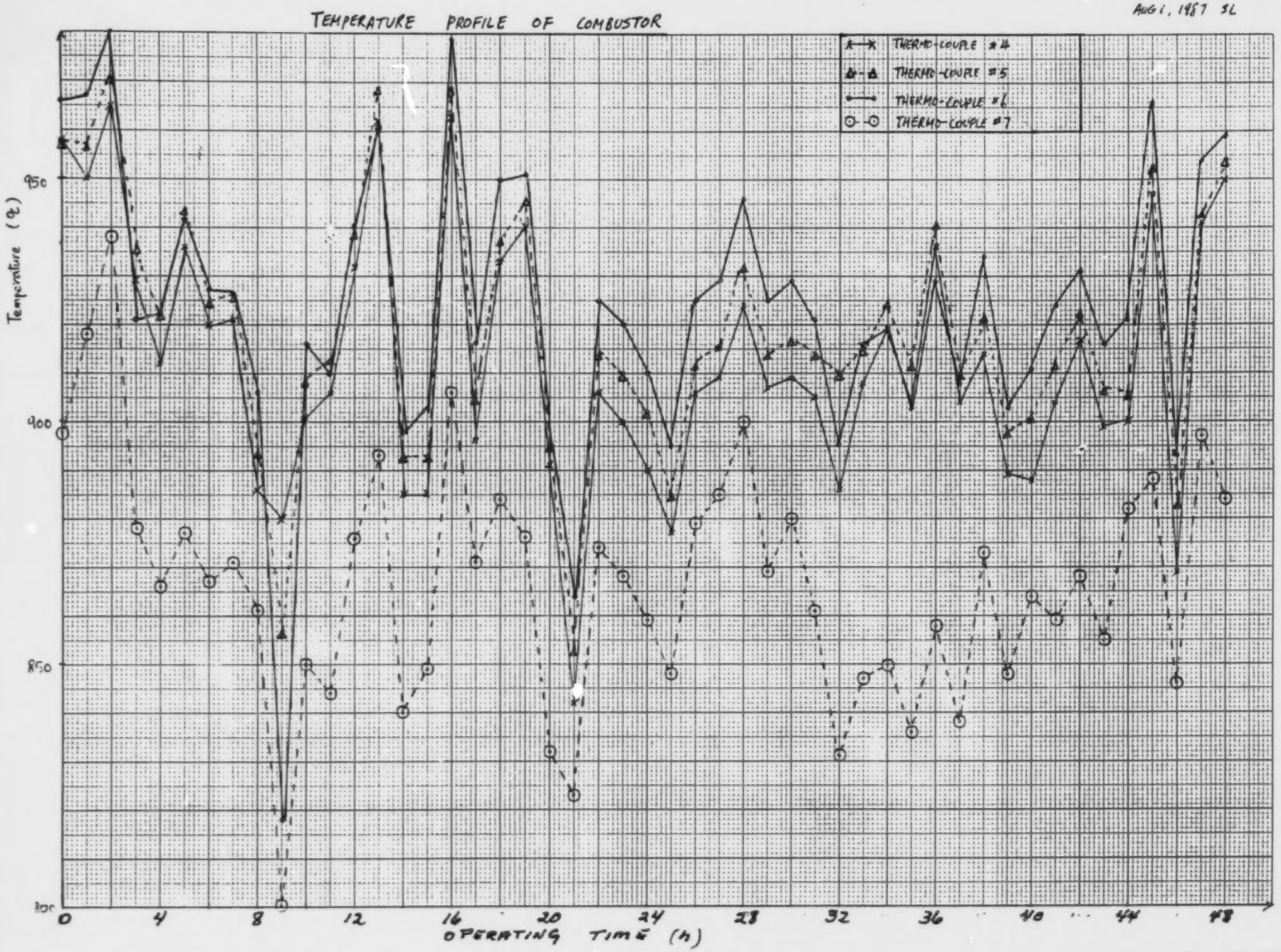
- the combustor volume is small relative to the surface area and mass of the pilot plant (a consequence of its smaller size). Thus bringing the unit up to operating temperature is slow, while it can cool rapidly if fuel feed is interrupted. Careful attention to the combustor was essential at all times. This was also the reason that a more volatile fuel like Forestburg coal was used at start up times to attain operating temperature with minimum delay. The Minto coal had proved inadequate to overcome heat loss in a preliminary test.

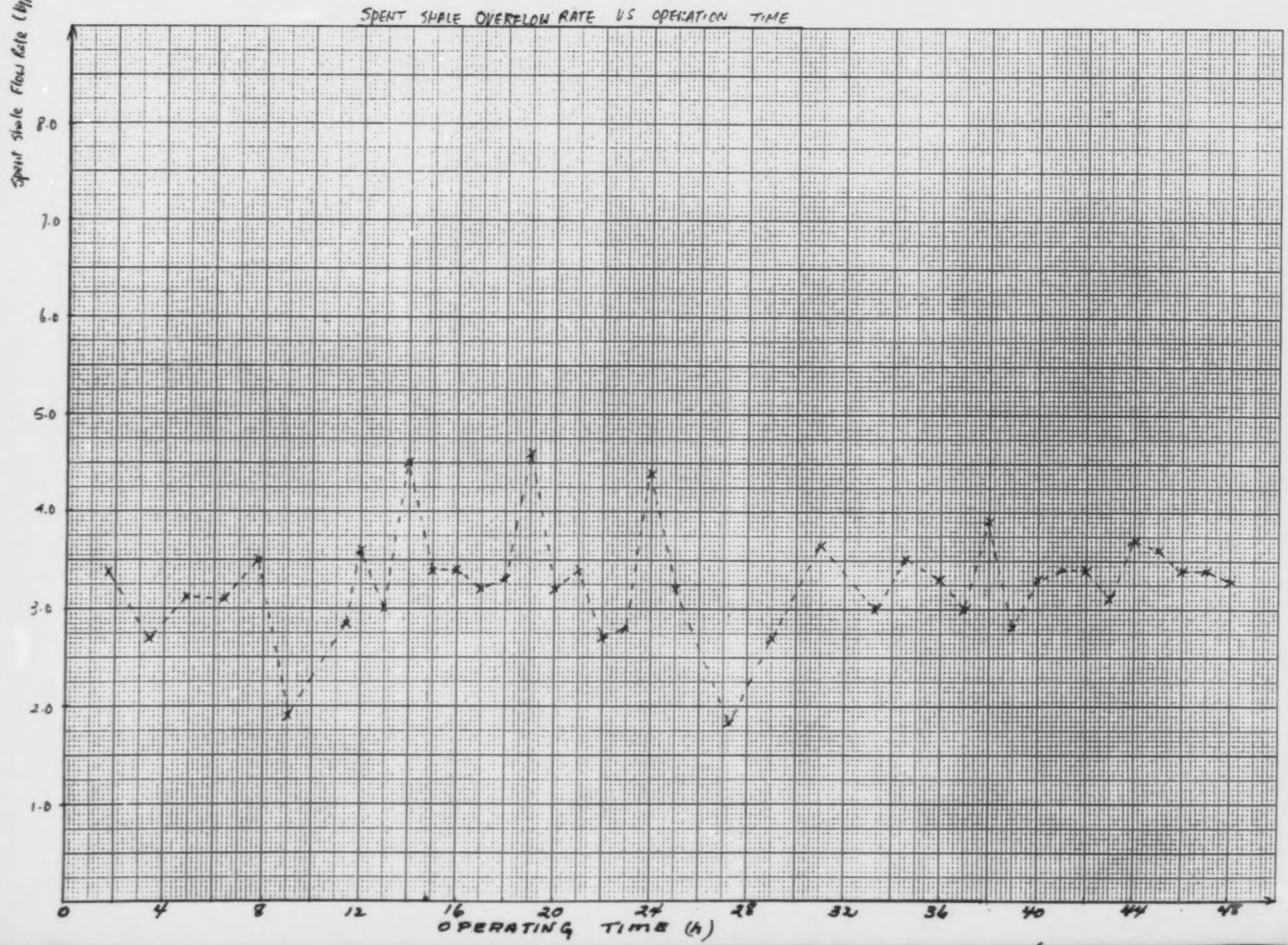
  It was found that a good guide to combustion rate and coal feed was oxygen content in the flue gas. A continuous O_2 monitor would be of great benefit to the unit's operators in providing a rapid indication of combustion problems or coal feed interruptions.

- a long test run should be preceded by several shorter runs to allow for the identification of, and correction of, potential operating problems. While this proceedure was followed by the Polymath/UBC team, such run-up tests were not specified in the contract nor was such time allowed for in the allotment of work hours. This need can be expected for most scaling-up tests and is raised as a point of practical knowledge gained.

- modification of existing compact arrangements of apparatus may not be simple if the benefits of the original apparatus setup are altered. Separation and redesign, using the existing components and new portions, may be more practical and time-saving. While this point may not apply generally it is likely to be the norm, and certainly raises the need to carefully evaluate the modifications that will be needed, at least to the extent of an isometric sketch or similar level, to be assured that the modifications will be manageable. In this case the problems arose from the very limited space within which piping reconnecting the combustor and retort and other additional items had to be accomodated.


TEMPERATURE PROFILE OF RETORT
AUG4, 1987 SL
Temperature (°C)
500
450
400
350
0
4
8
12
16
20
24
28
32
36
40
44
48
OPERATING TIME (h)
THERMO-COUPLE 14
THERMO-COUPLE 15
THERMO-COUPLE 16


TEMPERATURE PROFILE OF COMBUSTOR
AUG 1, 1987 SL
THERMO-COUPLE #4
THERMO-COUPLE #5
THERMO-COUPLE #6
THERMO-COUPLE #7
Temperature (°C)
950
900
850
800
0
4
8
12
16
20
24
28
32
36
40
44
48
OPERATING TIME (h)


SPENT SHALE OVERFLOW RATE VS OPERATION TIME
Spent Shale Flow Rate (lb/hr)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
4
8
12
16
20
24
28
32
36
40
44
48
OPERATING TIME (h)

7.0 RESULTS

7.1 General Comments on the Operating Mode

In Section 6.0 the performance of the experiment is described, and some of the operational changes and requirements commented on. These and other conditions that are considered relevant to the results obtained are further noted below:

a) Problems in maintaining a constant flow of spent shale from the retort overflow into the combustor dictated that spent shale be stored in a receiver after it left the retort, and that it be fed separately into the combustor. When fed it was usually cooled because of the delay. As discussed in Section 6.0 it was most convenient to take the spent shale from a previous part of the run, blend it with coal in the desired ratio, then feed the mixture into the combustor.

b) It was found necessary to operate the combustor at a large excess air level to provide transfer of heat to the recycled retort gases. The extra heat exchanger noted in Section 5.7 was required to obtain the necessary 580°C temperature in the feed gas to the retort.

c) The particularly low level of CaO in the spent shale resulted in operation at Ca/S molar levels in the combustor below unity. Thus the sulphur capture in the combustor was lower than anticipated.

d) Start up was with Forestburg coal, but by steady state the unit had been on Minto coal for some time. The small amount of Forestburg coal introduced during the experiment does not add any measureable dilution of the results attained.

7.2 Retorting Results

Operation of the retort is given by Tables 1 and 3. Table 2 gives the hourly data for a number of the retort variables. Chemical analysis of the cyclone solids and spent shale are presented in Tables 5.1 and 5.2.

Oil shale was fed to the retort at a rate of 3.78 kg/h. The fluidized bed was operated at a gas flow of 26.46 kg/h entering the retort at about 587°C. This flow gives a superficial gas velocity of 1.1 m/s. The oil shale was heated to a bed temperature of about 500°C, which was maintained over some 48 hours. During the first 18 hours the retort was at about 510°C, whereas during the last 15 hours it decreased to about 485°C.

The freeboard average temperature of 272°C was measured 0.8 m above the grid, whereas the bed depth was 0.36 m. The temperature was essentially constant as shown by Table 2, lines 6,7, and 8, which give the retorting temperature at different levels in the reactor.

Very little material was carried over from the fluid bed retort with the off-gas - Table 3 shows typical values of less than 0.025 kg/h or less than 1.0% of the feed. This result is somewhat surprising, but it may be related to the small amount of fines in the feed because of the double screening used in its preparation.

The elimination of carbon from the shale was below expectations. Table 5.1 shows that the carbon content of the retort overflow is 9.84%, whereas Appendix 1 shows the feed shale at 13.45%C (dry basis). The carbon elimination calculated from these two analyses, and using a silica and alumina balance to obtain an average weight loss for the shale, is only 24%. This suggests that the oil yield will be low, which indeed was found. Using the data in Table 6, the oil recovery as a percentage of the Fischer Assay is found as

$$\% \text{ Oil Recovery} = \frac{100 \text{ (Oil Recovered kg/h)}}{\text{Shale Feed Rate kg/h x Fischer Assay Oil Yield}}$$

$$= \frac{100\ (0.1339)}{3.783 \times 0.081} = 43.7\%$$

Table 5.5 shows the produced oil to have a specific gravity of 0.925, and contain 86.1% C, 11.6% H, 0.9% N, 0.6% S, and 0.8% O. The H/C mol ratio is thus 1.62. The boiling point data (given in Table 5.8) evaluated by simulated distillation show 10% distilled at about 278°C, and 50% distilled at 383°C. The samples collected in the demister had a somewhat higher boiling range than the condenser composite.

In previous studies with New Brunswick oil shale retorted in a spouted bed it was found [1] that the presence of spent shale in the reactor leads to low oil yields, presumably by promoting cracking or re-deposition of the oil on the spent shale. Thus in retorting beds containing about 1 kg spent shale and 5 kg sand, oil yields of about 90% of the Fischer Assay were delivered; whereas in a bed of 5 kg spent shale about 20% of the Fischer Assay was found. This effect has also been reported by Australian workers [2], and is thought to be related to species in the mineral content of particular shales which promote cracking. In the current test the steady state retorting bed contained about 6.4 kg, which with a feed rate of 3.78 kg/h gives a mean residence time of 1.7 hours for spent shale. Future work with fluidized bed retorting should emphasize lower mean residence times (or shallower beds) which should undoubtly improve oil yields.

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(1) - Tam, T. "Pyrolysis of Oil Shale in a Spouted Bed", M.A.Sc. Thesis, UBC, August 1987.

(2) - Levy,J.H., Mallon, R.G., and Wall, G.C., "Vapour phase cracking and coking of three Australian shale oils: kinetics in the presence and absence of shale ash", paper presented at Third Australian Workshop on Oil Shale, May, 1986.

Table 5.6 gives the analysis of the retort gases. These gases are essentially N_2, the carrier gas, as might be expected. The recirculated gas flow is about 26 kg/h, while the gas yield by Fischer Assay is 2.2%, which for a feed rate of 3.78 kg/h corresponds to 0.0832 kg/h gas produced by retorting under Fischer Assay conditions. The small potential gas yield, which corresponds to only 100 x (0.083) / 26.5 = 0.31% of the total gas flow, means that a net gas production rate is virtually impossible to measure. In the test there was a necessity to maintain a flow of "start-up" nitrogen through the unit because of downstream losses. This flow amounted to about 20% of the total recirculating gas. The primary species generated are CO_2, CO, H_2, and C_3's.

Material and Enthalpy balances around the retorting system are summarized in Tables 6 and 7. The average mass lost from the shale by the mass rate measurement of solids is 15% of the solids input weight. The Fischer Assay gives precisely this percentage weight loss where all oil, water, and gas are produced. As discussed above only 44% of the Fischer Assay oil was recovered.

The retorting gases are recycled after cooling to about 48°C in heat exchanger E-1. In this experiment a large cooling water flow (1200 kg/h of cooling water versus 26.5 kg/h of gas flow) meant that no measureable temperature rise occurred for the water in the exchangers, whereas the gas temperature dropped from 136°C to 48°C. The recirculating gas was heated up somewhat in the large recirculation gas blower. This unit had a recycle (Figure 1) because it was oversized, which resulted in the net outlet gas increasing in temperature from 48°C to about 100°C by frictional heating.

After the input of cooler "start up" nitrogen, the retort recycle gas was heated from about 83°C to 587°C (Table 7) for the retort inlet by exchange with the combustor flue gas.

7.3 **Combustor Results**

The fluid bed combustor was operated at a high superficial velocity of about 3 m/s. Ash left the combustor almost equally split between the bed overflow (0.9 kg/h) and carryover to the cyclone (1.2 kg/h). Data are given in Tables 1, 2, 3, 5.3, 5.4, and 5.7. Coal was fed at 2.37 kg/h with spent shale containing 9%C at 2.03 kg/h. The air rate was very high at a value of 57.7 kg/h which corresponds to an excess air level of about 120%. The O_2/C mol ratio was about 2.74, which yielded a flue gas containing 11.6% O_2. The carbon level in the cyclone product was 4.7% (Table 5.4), compared to 0.35% in the bed overflow (Table 5.3). These analyses and flowrates indicate a carbon burnout based on solids analyses alone of 97.0%.

Sulphur was captured via the reaction of the CaO in the spent shale. Unfortunately the spent shale feed rate at 2.03 kg/h was insufficient to achieve a Ca/S mol ratio in excess of unity. The off-gas contained 1300 ppmv SO_2 (Table 5.7), and 140 ppm NO_x. The coal and shale bring 0.1616 kgS/h to the combustor, and the percentage S capture based on the gas concentrations is 45%, at a Ca/S mol ratio of 0.52.

Measurement of SO_2 levels in the combustor flue gas prior to feeding spent shale gave values of 1830 ppm. Calculations by sulphur balance show that if all the entering coal sulphur were burnt to SO_2 and none captured by coal ash the concentration of SO_2 would be 2440 ppm for the combustion conditions used.

7.4 Achieving Operational Objectives

All the basic objectives stated in Section 3.0, Test Objectives were met. The integrated system of pyrolysis reactor and combustion unit operated together over a steady state run of 48 hours. During this steady state period the only fuels used were the New Brunswick oil shale and Minto coal, except for start up and the one difficulty described. All gas and liquid streams and samples of solids specified were collected and analysed.

Secondary operational points that were to be implemented during the experiment were successful in part, while adjustments particular to the apparatus determined the actual running modes. For example, the successful points met were:

- the retort used only shale, and contained no sand;

- the combustor was started with an initial sand bed and using a high volatile Forestburg coal, but after stable temperature was reached only Minto coal and spent shale were used. The combustor solids bed, particularly towards the latter part of the experiment, may be considered to contain no other material;

- the residence time for solids in the combustor and retort was calculated from experimental data;

- all liquid, gas and solids streams were collected and measured as specified. Losses for gas and liquid streams occurred as described in Section 6, but were either unmeasureable or of a low value.

A less successful secondary objective was to maintain the retort cyclone at the retort temperature. Heat loss from the cyclone and the interconnecting piping determined the temperature attained. This temperature can be seen from Table 2 to be reasonably steady. As the experiment progressed the temperature difference between the retort and cyclone decreased, till it was at a relatively stable level for the last third of the run.

The combustor was not operated with 5% excess air. The requirements for fluidization and combustion, and for heat transfer through two gas to gas heat exchangers to the retort gas, were determining factors for the air level used. This was much larger than anticipated. High excess air was also required to prevent sintering in the bed itself.

7.5 **Tables of Results**

Results of the experiment are shown fully in the accompanying tables:

Table 1 gives the main operating conditions.

Table 2 lists a series of measurements made at one hour intervals for specified items.

Table 3 gives a listing of specific measurements relating to solids and liquid streams at 6 hour intervals.

Table 4 gives the size distribution of oil shale and Minto coal, and for the spent solids of the combustor and retort.

Tables 5.1 to 5.8 inclusive give the results of analyses of solid, liquid and gaseous samples taken durig the experiment.

Appendix 5 lists the methods of analytical determination used.

7.6 **Recommendations for Further Work**

Following up indications noted in the present experiment, future work could benefit from:

- runs at lower bed depths to give shorter residence times for shales in the retort;

- runs at higher spent shale rate to improve sulphur capture;

- improvements in the heat recovery systems to permit greater shale throughputs and operation at lower excess air in the combustor;

- future work at the UBC unit could have the GC system for CO2 modified to use thermal conductivity detectors and obtain improved data. The problems with sulphur balance could also be corrected.

TABLE 1 - OPERATING CONDITIONS

Primary Operating Conditions		
Oil Shale Feed Rate	P1	3.78 kg/h
Retort Temperature, top *	TI-1	272 °C
Retort Temperature, bed	TI-2	497 °C
Coal Feed Rate	C1	2.37 kg/h
Combustor Temperature, top *	TI-3	866 °C
Combustor Temperature, bed	TI-4	911 °C
Secondary Operating Conditions		
Solids Residence Time in Retort		1.70 h
Gas Residence Time in Retort **		0.88 s
Retort Gas Recycle Rate	FI-3	26.46 kg/h
Solids Mean Residence Time in Combustor		0.66 h
Gas Residence Time in Combustor **		0.33 s
Air Flow to Combustor	FI-1	57.7 kg/h

* Freeboard

** Based on Empty Reactor

TABLE 2.0 - EXPERIMENTAL MEASUREMENT AT 1 HOUR INTERVALS

Line No.	Item	Symbol	Units
1	Oil Shale Feed Rate	P1	kg/h
2	Recycle Retort Gas Flow Rate	FI-3	kg/h
3	Start-up N2 for Pyrolyser	FI-5	kg/h
4	Net Retort Gas Flow Rate (negliibly small for closed system)	FI-2	kg/h
5	Retort Top Temperature (T.C.#18) 32 inches above the grid	TI-1	^{o}C
6	Retort Bottom Temperature (T.C.#14) 2 inches above the grid	TI-2	^{o}C
7	Retort Intermediate Temperature (T.C.#15) see Figure 1	TI-12	^{o}C
8	Retort Intermediate Temperature (T.C.#16) see Figure 1	TI-13	^{o}C
9	Retort Cyclone Temperature	TI-5	^{o}C
10	Temperature of Cooled Retort Fluids	TI-6	^{o}C
11	Cooling Water Discharge Temperature from Exchanger	TI-7	^{o}C
12	Temperature of Cold Retort Recycle Gas	TI-8	^{o}C
13	Temperature of Hot Retort Recycle Gas	TI-9	^{o}C
14	Temperature of Cold Flue Gas	TI-10	^{o}C
15	Combustor Cyclone Temperature	TI-11	^{o}C
16	Combustor Top Temperature (T.C.#7) 24.5 inches above the grid	TI-3	^{o}C
17	Combustor Bottom Temperature (T.C.#4) 4.5 inches above the grid	TI-4	^{o}C
18	Coal Feed Rate (as recieved basis)	C1	kg/h
19	Flue Gas Flow Rate (by calculation, data in Tables 5.3 and 5.7)	FI-4	kg/h
20	Combustor Air Flow Rate	FI-1	kg/h

TABLE 2.1 - EXPERIMENTAL MEASUREMENT AT 1 HOUR INTERVALS (DATA)

Line	Symbol	TIME INTO RUN (HOURS) 1	2	3	4	5	6	7	8	Units
1	P1	3.783	3.783	3.783	3.783	3.783	3.783	3.783	3.783	kg/h
2	FI-3	25.15	26.93	26.85	27.45	26.85	27.45	23.65	23.80	kg/h
3	FI-5	6.25	5.07	4.55	4.55	4.55	4.55	4.55	4.40	kg/h
4	FI-2	----------negligibly small, due to leakage ----------								kg/h
5	TI-1	324	333	331	327	325	319	308	301	°C
6	TI-2	506	515	523	520	517	515	516	512	°C
7	TI-12	432	451	469	496	511	513	509	505	°C
8	TI-13	390	415	426	424	438	486	520	522	°C
9	TI-5	157	168	168	169	167	169	152	150	°C
10	TI-6	63	66	68	67	66	66	57	55	°C
11	TI-7	13	13	13	13	13	13	13	13	°C
12	TI-8	414	421	420	406	395	388	403	412	°C
13	TI-9	597	606	600	583	579	571	585	585	°C
14	TI-10	633	640	634	617	616	609	623	621	°C
15	TI-11	783	795	780	758	764	757	763	751	°C
16	TI-3	918	938	878	866	877	867	871	861	°C
17	TI-4	950	965	929	912	936	920	921	886	°C
18	C1	2.375	2.375	2.375	2.375	2.375	2.375	2.375	2.375	kg/h
19	FI-4	58.61	58.61	58.61	58.61	58.61	58.61	58.61	58.61	kg/h
20	FI-1	57.68	57.68	57.68	57.68	57.68	57.68	57.68	57.68	kg/h

TABLE 2.2 - EXPERIMENTAL MEASUREMENT AT 1 HOUR INTERVALS (DATA)

Line	Symbol	TIME INTO RUN (HOURS) 9	10	11	12	13	14	15	16	Units
1	P1	3.783	3.783	3.783	3.783	3.783	3.783	3.783	3.783	kg/h
2	FI-3	25.70	23.65	23.65	23.05	23.05	23.05	26.85	21.35	kg/h
3	FI-5	4.30	4.55	4.55	4.55	4.55	4.55	4.55	6.25	kg/h
4	FI-2	----------negligibly small, due to leakage ----------								kg/h
5	TI-1	305	292	415	284	284	286	285	278	^{o}C
6	TI-2	453	483	489	502	513	510	507	510	^{o}C
7	TI-12	461	479	482	494	504	504	501	501	^{o}C
8	TI-13	469	488	495	509	519	512	516	509	^{o}C
9	TI-5	165	151	145	144	142	143	140	140	^{o}C
10	TI-6	61	54	51	50	49	49	49	51	^{o}C
11	TI-7	13	13	13	13	13	13	13	13	^{o}C
12	TI-8	269	363	382	408	427	403	396	389	^{o}C
13	TI-9	461	544	564	594	608	581	574	577	^{o}C
14	TI-10	512	583	602	633	646	613	607	614	^{o}C
15	TI-11	684	729	735	764	778	736	733	763	^{o}C
16	TI-3	789	850	844	876	893	840	849	906	^{o}C
17	TI-4	870	901	906	932	962	885	885	963	^{o}C
18	C1	2.375	2.375	2.375	2.375	2.375	2.375	2.375	2.375	kg/h
19	FI-4	58.61	58.61	58.61	58.61	58.61	58.61	58.61	58.61	kg/h
20	FI-1	57.68	53.79	53.79	57.68	57.68	53.79	53.79	53.79	kg/h

TABLE 2.3 - EXPERIMENTAL MEASUREMENT AT 1 HOUR INTERVALS (DATA)

Line	Symbol	TIME INTO RUN (HOURS) 17	18	19	20	21	22	23	24	Units
1	P1	3.783	3.783	3.783	3.783	3.783	3.783	3.783	3.783	kg/h
2	FI-3	23.99	23.99	21.07	21.07	21.07	20.65	21.07	21.50	kg/h
3	FI-5	4.21	4.21	7.13	7.13	7.13	7.55	7.13	6.70	kg/h
4	FI-2	----------negligibly small, due to leakage----------								kg/h
5	TI-1	278	281	245	243	235	249	229	231	°C
6	TI-2	510	515	497	503	500	481	478	484	°C
7	TI-12	504	511	481	491	490	470	466	473	°C
8	TI-13	509	522	469	485	495	416	457	489	°C
9	TI-5	141	143	113	115	107	130	117	119	°C
10	TI-6	50	52	39	40	37	43	40	41	°C
11	TI-7	13	13	13	13	13	13	13	13	°C
12	TI-8	397	398	416	416	420	454	425	427	°C
13	TI-9	582	583	585	585	579	585	588	588	°C
14	TI-10	617	618	624	620	616	628	632	631	°C
15	TI-11	753	757	755	733	727	755	753	747	°C
16	TI-3	871	884	876	832	823	874	868	859	°C
17	TI-4	896	933	940	891	842	906	900	890	°C
18	C1	2.375	2.375	2.375	2.375	2.375	2.375	2.375	2.375	kg/h
19	FI-4	58.61	58.61	58.61	58.61	58.61	58.61	58.61	58.61	kg/h
20	FI-1	53.79	53.79	53.79	56.90	53.79	57.68	57.68	57.68	kg/h

TABLE 2.4 - EXPERIMENTAL MEASUREMENT AT 1 HOUR INTERVALS (DATA)

Line	Symbol	TIME INTO RUN (HOURS) 25	26	27	28	29	30	31	32	Units
1	P1	3.783	3.783	3.783	3.783	3.783	3.783	3.783	3.783	kg/h
2	FI-3	23.20	23.20	23.99	23.99	20.74	20.45	18.05	18.05	kg/h
3	FI-5	5.00	5.00	4.21	4.21	4.21	3.95	3.95	3.95	kg/h
4	FI-2	----------negligibly small, due to leakage----------								kg/h
5	TI-1	256	246	303	285	272	270	266	256	°C
6	TI-2	498	490	515	503	506	503	503	493	°C
7	TI-12	491	481	491	488	498	496	484	485	°C
8	TI-13	499	482	484	492	508	511	491	501	°C
9	TI-5	144	130	167	150	144	143	139	130	°C
10	TI-6	51	44	55	51	51	49	46	44	°C
11	TI-7	13	13	13	13	13	13	13	13	°C
12	TI-8	409	430	428	434	440	436	434	435	°C
13	TI-9	587	594	597	604	605	603	596	591	°C
14	TI-10	619	638	639	648	646	647	640	633	°C
15	TI-11	742	758	765	774	764	766	755	741	°C
16	TI-3	848	879	885	900	869	880	861	831	°C
17	TI-4	877	906	909	924	907	909	905	886	°C
18	C1	2.375	2.375	2.375	2.375	2.375	2.375	2.375	2.375	kg/h
19	FI-4	58.61	58.61	58.61	58.61	58.61	58.61	58.61	58.61	kg/h
20	FI-1	57.68	57.68	57.68	57.68	57.68	57.68	57.68	57.68	kg/h

TABLE 2.5 - EXPERIMENTAL MEASUREMENT AT 1 HOUR INTERVALS (DATA)

Line	Symbol	TIME INTO RUN (HOURS) 33	34	35	36	37	38	39	40	Units
1	P1	3.783	3.783	3.783	3.783	3.783	3.783	3.783	3.783	kg/h
2	FI-3	19.15	18.55	18.15	17.79	16.89	18.99	19.69	18.99	kg/h
3	FI-5	3.85	4.05	3.85	4.21	4.21	4.21	4.21	4.21	kg/h
4	FI-2	----------negligibly small, due to leakage----------								kg/h
5	TI-1	257	255	252	249	245	251	254	255	°C
6	TI-2	492	491	489	488	485	490	491	487	°C
7	TI-12	485	482	480	478	478	481	485	479	°C
8	TI-13	501	499	497	494	494	494	500	495	°C
9	TI-5	133	132	129	126	124	131	134	135	°C
10	TI-6	45	45	44	43	42	45	46	46	°C
11	TI-7	13	13	13	13	13	13	13	13	°C
12	TI-8	432	435	433	436	434	436	437	425	°C
13	TI-9	591	595	589	596	588	593	596	585	°C
14	TI-10	635	639	634	646	633	636	638	629	°C
15	TI-11	747	752	744	760	742	752	747	743	°C
16	TI-3	847	850	836	858	838	873	848	864	°C
17	TI-4	908	919	904	936	904	914	889	888	°C
18	C1	2.375	2.375	2.375	2.375	2.375	2.375	2.375	2.375	kg/h
19	FI-4	58.61	58.61	58.61	58.61	58.61	58.61	58.61	58.61	kg/h
20	FI-1	57.68	57.68	57.68	57.68	57.68	57.68	57.68	57.68	kg/h

TABLE 2.6 - EXPERIMENTAL MEASUREMENT AT 1 HOUR INTERVALS (DATA)

Line	Symbol	TIME INTO RUN (HOURS) 41	42	43	44	45	46	47	48	Units
1	P1	3.783	3.783	3.783	3.783	3.783	3.783	3.783	3.783	kg/h
2	FI-3	18.99	18.99	18.90	18.30	17.80	17.45	16.75	16.75	kg/h
3	FI-5	4.21	4.21	4.30	4.30	4.40	4.55	5.25	5.25	kg/h
4	FI-2	----------negligibly small, due to leakage----------								kg/h
5	TI-1	253	249	246	242	241	236	226	224	°C
6	TI-2	486	485	486	485	486	485	482	483	°C
7	TI-12	478	476	476	475	476	475	470	470	°C
8	TI-13	494	492	493	491	494	494	488	470	°C
9	TI-5	132	129	127	123	122	118	111	109	°C
10	TI-6	44	44	43	42	41	40	38	37	°C
11	TI-7	13	13	13	13	13	13	13	13	°C
12	TI-8	426	429	435	440	449	447	448	452	°C
13	TI-9	585	588	591	595	606	595	604	607	°C
14	TI-10	630	635	637	643	657	640	657	660	°C
15	TI-11	745	750	747	755	773	743	776	776	°C
16	TI-3	859	868	855	872	898	846	897	884	°C
17	TI-4	904	917	899	900	947	869	941	950	°C
18	C1	2.375	2.375	2.375	2.375	2.375	2.375	2.375	2.375	kg/h
19	FI-4	58.61	58.61	58.61	58.61	58.61	58.61	58.61	58.61	kg/h
20	FI-1	57.67	57.68	57.68	57.68	57.68	57.68	57.68	57.68	kg/h

TABLE 3 - EXPERIMENTAL MEASUREMENTS AT 6 HOUR INTERVALS

Meas. Point	Item	TIME INTO RUN (HOURS) (All measurements given in kg/h) 6	12	18	24	30	36	42	48
P2	Spent Shale Rate from Retort	3.071	3.050	3.467	3.517	2.360	3.342	3.300	3.412
P3	Retort Cyclone Solids	0.003	0.014	0.025	0.000	0.000	0.000	0.002	0.000
P4	Kerogen Rate	0.151	0.093	0.160	0.146	0.157	0.119	0.115	0.127
C2	Coal Ash Solids	0.678	0.784	0.700	0.675	0.850	1.327	1.046	1.083
C3	Combustor Cyclone Solids	1.417	1.300	0.867	1.117	1.550	1.100	0.917	1.017

TABLE 4.1 - SIZE DISTRIBUTION OF OIL SHALE AND COAL

OIL SHALE			MINTO COAL		
Screen No. US Series	di (mm)	Xi Weight Fraction	Screen No. US Series	di (mm)	Xi Weight Fraction
			+ 8	4.76	0.0533
- 4 + 8	3.57	0.3371	- 4 + 8	3.57	0.2307
- 8 + 16	1.79	0.2259	- 8 + 16	1.79	0.2793
- 16 + 30	0.89	0.1240	- 16 + 30	0.89	0.1757
- 30 + 50	0.444	0.0790	- 30 + 50	0.444	0.1053
- 50 + 100	0.223	0.0634	- 50 + 100	0.223	0.0763
- 100 + 200	0.11	0.0463	- 100 + 200	0.11	0.0327
- 200	0.037	0.1243	- 200	0.037	0.0467
	$d = \sum Xi.di = 1.77$ mm			$d = \sum Xi.Di = 1.80$ mm	

TABLE 4.2 - SIZE DISTRIBUTION OF RETORT SPENT SHALES AND COMBUSTOR PRODUCTS

RETORT SPENT SHALE			RETORT CYCLONE		
Screen No. US Series	di (mm)	Xi Weight Fraction	Screen No. US Series	di (mm)	Xi Weight Fraction
- 4 + 8	3.57	0.004			
- 8 + 16	1.79	0.667			
- 16 + 30	0.89	0.317			
- 30 + 50	0.444	0.008	- 30 + 50	0.444	0.057
- 50 + 100	0.223	0.001	- 50 + 100	0.223	0.136
- 100	0.075	0.003	- 100 + 200	0.111	0.104
			- 200	0.037	0.703
	d = 1.49 mm			d = 0.093 mm	
COMBUSTOR BED MATERIAL			COMBUSTOR CYCLONE		
- 4 + 8	3.57	0.020			
- 8 + 16	1.79	0.776	- 8 + 16	1.79	0.177
- 16 + 30	0.89	0.192	- 16 + 30	0.89	0.340
- 30 + 50	0.444	0.005	- 30 + 50	0.444	0.069
- 50 + 100	0.223	0.002	- 50 + 100	0.223	0.059
- 100	0.075	0.005	- 100 + 200	0.111	0.066
			- 200	0.037	0.289
	d = 1.63 mm			d = 0.68 mm	

TABLE 5.1 - CHEMICAL ANALYSIS OF RETORT SPENT SHALE *

ANALYSIS ITEM	TIME INTO RUN (HOURS) 7:45	13	19	25	31	37	43	48	Avg.
%C	9.74	10.60	8.92	8.08	9.10	10.60	10.70	11.00	9.84
%S	0.28	0.29	0.31	0.32	0.29	0.32	0.31	0.32	0.30
%Ash	84.70	83.48	85.05	86.02	84.68	82.55	82.31	81.94	83.84
Mineral Matter									
$\%SiO_2$	62.33	63.53	62.73	63.23	63.25	63.45	63.59	63.01	63.14
$\%Al_2O_3$	15.76	15.88	15.28	15.87	15.65	15.85	15.94	15.69	15.74
$\%Fe_2O_3$	6.83	6.42	6.31	6.41	6.45	6.41	6.52	6.49	6.48
%MgO	2.15	2.18	2.16	2.19	2.19	2.17	2.23	2.20	2.18
%CaO	4.54	4.95	5.54	5.46	5.17	5.16	5.18	5.25	5.16

* Carbon determined separately, remainder by Plasma Spectroscopy.

For additional constituents of mineral matter analysis see Appendix 2.

TABLE 5.2 - CHEMICAL ANALYSIS OF RETORT CYCLONE SOLIDS*

ANALYSIS ITEM	TIME INTO RUN (HOURS) 1:45	2:15	7	13	19	25	31	37	43	48	Avg.
%C	7.26	9.77	9.52	11.60	12.90	14.20	9.59	13.20	14.90	13.10	11.60
%S	0.28	0.44	0.37	0.35	0.37	0.41	0.43	0.44	0.40	0.40	0.39
%Ash	88.60	85.56	84.42	81.76	80.07	78.16	85.34	80.06	77.86	80.36	82.22
Mineral Matter											
$\%SiO_2$	63.39	64.41	63.67	61.63	59.82	62.50	61.53	59.85	59.60	58.42	61.48
$\%Al_2O_3$	15.60	13.52	14.82	15.64	15.88	16.17	15.82	16.09	16.43	14.49	15.45
$\%Fe_2O_3$	6.95	7.19	6.83	7.98	9.83	8.60	8.33	8.60	10.23	8.25	8.28
%MgO	2.19	1.86	1.98	2.05	2.09	2.12	2.06	2.08	2.08	1.80	2.03
%CaO	4.52	4.72	4.60	3.43	3.40	3.23	3.65	3.24	3.19	2.99	3.70

* Carbon determined separately, remainder by Plasma Spectroscopy

For additional constituents of mineral matter analysis see Appendix 2.

TABLE 5.3 - CHEMICAL ANALYSIS OF COMBUSTOR BED MATERIAL (SPENT SOLIDS)*

ANALYSIS ITEM	TIME INTO RUN (HOURS) 2:45	7	13	19	25	31	37	43	48	Avg.
%C	1.73	0.32	0.20	0.23	0.15	0.23	0.32	0.28	1.44	0.54
%S	0.36	0.65	0.64	0.77	0.85	0.84	0.78	0.91	0.72	0.72
%Ash	98.04	99.77	99.89	99.75	99.75	99.76	99.66	99.66	98.25	99.39
Mineral Matter										
$\%SiO_2$	77.00	62.16	61.98	61.31	61.48	61.24	61.45	60.23	60.76	61.33**
$\%Al_2O_3$	8.57	14.88	15.87	15.36	14.97	15.46	15.24	14.73	14.93	15.18**
$\%Fe_2O_3$	6.27	8.84	8.44	8.31	8.00	8.49	8.42	8.09	8.33	8.37**
%MgO	1.09	1.97	2.02	2.01	1.96	2.09	2.08	2.02	2.05	2.03**
%CaO	3.33	4.57	4.54	5.18	5.28	5.71	5.41	5.82	5.29	5.23**

* Carbon determined separately, remainder by Plasma Spectroscopy. ** excludes sample at 2:45 h.

For additional constituents of mineral matter analysis see Appendix 2.

TABLE 5.4 - CHEMICAL ANALYSIS OF COMBUSTOR CYCLONE SOLIDS *

ANALYSIS ITEM	TIME INTO RUN (HOURS) 1:30	7	13	19	25	31	37	43	48	Avg.
%C	5.23	3.07	5.74	5.21	5.53	3.48	5.00	4.42	5.10	4.75
%S	0.29	0.473	0.62	0.63	0.69	0.67	0.57	0.61	0.57	0.57
%Ash	94.52	96.78	93.75	94.43	94.16	96.40	94.72	95.28	94.68	94.97
Mineral Matter										
$\%SiO_2$	75.37	57.64	53.37	54.33	55.50	56.13	52.90	55.57	53.48	54.87**
$\%Al_2O_3$	5.72	12.59	13.65	14.06	14.43	14.01	13.99	14.26	13.75	13.84**
$\%Fe_2O_3$	11.97	15.05	18.11	17.15	16.30	14.15	19.72	16.93	19.84	17.16**
%MgO	0.52	1.58	1.56	1.66	1.72	1.82	1.50	1.69	1.55	1.64**
%CaO	2.54	4.64	4.68	4.59	4.54	5.13	4.65	4.46	4.65	4.67**

* Carbon determined separately, remainder by Plasma Spectroscopy. ** excludes sample at 1:30 h.

For additional constituents of mineral matter analysis see Appendix 2.

TABLE 5.5 - COMPOSITION AND GRAVITY OF RETORT LIQUID

Shale Oil Sample Time into run (h)	Specific Gravity 60oF/60oF	Weight % Composition (dry ash free) C	H	N	S	O	Ash content wt% (as received)
7	0.9372	86.54	11.68	0.99	0.62	0.17	4.80
13	0.9360	85.87	11.73	0.91	0.77	0.72	3.37
19	0.9306	85.55	11.58	0.91	0.70	1.26	2.41
25	0.9377	86.36	11.90	0.98	0.61	0.15	3.46
31	0.9376	85.00	11.43	0.97	0.86	1.74	3.51
37	0.9295	85.79	11.64	0.89	0.51	1.17	2.67
43	0.9249	86.32	11.85	0.83	0.50	0.50	1.25
48	0.9234	86.15	11.82	0.78	0.50	0.75	1.81
Condenser composite	0.9138	86.20	11.59	0.84	0.54	0.83	2.23
Weighted Average	0.9246	86.05	11.64	0.89	0.60	0.82	2.87

TABLE 5.6 - RETORT GAS ANALYSIS (% VOLUME)

Time into Run (h)		1	7.25	13	20	24.6	31.03	36.92	41.93	46.08	Average (std.dev.±)
Hydrogen,	ppm	1,463	1,484	338	1,292	1,716	2,199	1,063	1,280	1,084	1,324 (497)
Oxygen,	%	0.59	0.59	1.32	3.18	0.37	0.61	1.03	1.49	1.37	1.17 (0.81)
Nitrogen	%	----------	----------	balance	----------	----------	----------	----------	balance	----------	----------
CO_2	%	1.70	2.86	5.37	1.89	2.06	1.39	2.28	0.97	1.23	2.19 (1.25)
CO	ppm	1,545	2,125	4,446	2,708	1,224	1,836	2,160	1,613	0	2,193 (914)
CH_4	ppm	926	849	767	771	729	1,182	671	629	680	800 (160)
C_2H_6	ppm	266	259	216	262	761	417	157	165	296	311 (175)
C_2H_4	ppm	290	205	369	265	285	421	323	269	659	343 (126)
C_2H_2	ppm	0	0	148	154	139	0	0	29	287	151 (82)
C_3's	ppm	666	710	667	1,745	1,049	2,527	1,017	1,214	1,631	1,247 (586)
C_4's	ppm	490	246	813	1,364	1,046	1,624	180	610	320	744 (481)
H_2S	ppm	14	3	0	0	44	0	0	2	2	7.2(13.7)
SO_2	ppm	0	0	0	0	0	6	10	0	0	1.8 (3.5)
NO_x	ppm	----------	----------	----------	See note below	----------	----------	----------	----------	----------	-

Note: NO_x is not anticipated within the retort since no oxygen enters, no combustion occurs, and the temperature is below 500°C.

TABLE 5.7 - COMBUSTOR FLUE GAS ANALYSIS (BY VOLUME, DRY BASIS)

Analysis Item		TIME INTO RUN (HOURS) 1	7.33	13.0	20	24.5	31.25	36.88	42.02	46.17	Average (std.dev.±)
Oxygen	%	10.15	12.17	13.26	9.41	9.60	12.82	12.70	12.00	12.52	11.63 (1.40)
Nitrogen	%	----------balance----------					----------balance----------				
CO_2	%	9.50	7.20	5.80	6.82	6.90	5.23	6.95	5.50	8.35	6.92 (1.29)*
CO	ppm	131	97	52	0	180	127	208	130	757	187 (210)
CH_4	ppm	0	0	0	0	0	0	0	0	0	0
SO_2	ppm	1,788	851	1,693	1,384	1,368	516	1,196	1,430	1,569	1,311 (383)
NO_x	ppm	125	142	159	135	130	125	160	130	135	138 (13)

* Calculated carbon mass balance gives average 7.7 %.

FLUE GAS CALCULATION

Air to combustor = 57.7 kg/h, hence N_2 in combustion air = 0.767 x 57.7 = 44.26 kg/h
Mols N_2 in combustion air = 44.26 / 28 = 1.5807 k.mol/h .
From above the Dry Flue Gas Composition (average) has 11.63% O_2, 6.92% CO_2, 0.13% SO_2
N_2 in Flue Gas = 100 - (11.63 + 6.92 + 0.13) = 81.32% N_2
Thence Mols Dry Flue Gas by nitrogen balance = 1.5807 / 0.8132 = 1.9438 k.mol/h
Molecular weight Dry Flue Gas = 0.8132 (28) + 0.1163 (32) + 0.0692 (44) + 0.0013 (64)
= 29.61 kg/k.mol
Dry Flue Gas Flow Rate = 29.61 (1.9438) = 57.56 kg/h
Wet Flue Gas Flow Rate = 58.61 kg/h

TABLE 5.8 - BOILING POINT CURVES FOR OIL SAMPLES

Time into Run (h)	7	13	19	25	31	37	43	48	Cond. Comp.* Samp.
Percent of	Boiling Point (Deg. C)								
IBP	161.7	163.7	215.5	32.3	173.3	179.1	185.1	214.5	142.0
5	235.7	237.2	272.1	252.9	243.5	250.2	254.6	271.9	185.8
10	269.5	270.5	296.0	278.3	269.4	270.9	276.3	294.7	206.7
15	291.2	293.1	311.1	295.9	288.7	288.3	291.6	306.3	221.7
20	307.4	309.2	328.3	308.8	301.5	298.7	302.1	319.6	236.2
25	326.2	328.3	338.8	324.0	315.6	308.2	312.5	333.2	252.0
30	339.0	340.2	351.0	336.6	331.4	319.1	322.4	340.8	267.9
35	353.3	355.4	366.0	348.4	340.6	331.9	333.5	351.1	287.6
40	370.2	372.3	382.1	364.7	354.0	338.6	339.8	362.7	303.0
45	386.7	391.3	396.2	382.0	369.9	346.2	347.0	376.5	324.8
50	401.6	403.6	407.5	396.7	385.6	358.0	358.1	390.9	341.5
55	412.5	415.1	419.2	408.1	401.1	370.7	369.7	400.9	360.8
60	425.2	427.7	430.5	420.1	412.0	383.4	381.3	410.5	383.2
65	437.7	440.5	442.3	432.5	424.8	395.6	393.6	420.0	401.1
70	450.0	453.8	456.3	445.4	438.8	404.9	402.7	430.4	414.9
75	470.7	475.3	477.4	463.8	453.4	415.7	411.7	441.9	429.1
80	493.4	497.8	499.1	488.4	478.8	426.9	421.7	455.0	444.4
85	512.3	516.1	517.1	510.3	504.3	440.4	433.2	476.6	467.3
90	529.2	532.6	533.0	529.6	526.1	458.3	447.0	498.5	498.0
95	547.8	555.0	556.3	552.6	546.7	498.7	474.0	516.2	525.2
FBP	596.9	611.7	612.2	611.3	602.2	548.6	515.0	532.4	559.8
				D-86 CORRELATION					
IBP	259.9	261.2	294.1	220.0	263.9	266.8	272.0	293.0	216.5
10	307.1	308.4	330.8	313.0	305.0	304.9	309.5	326.9	243.5
20	331.3	332.6	348.7	332.4	326.0	319.5	323.2	341.0	263.3
30	352.6	354.1	365.5	350.7	343.7	330.8	333.4	354.5	285.2
50	396.8	398.8	402.6	392.1	381.2	354.3	354.3	386.4	338.2
70	441.5	445.1	447.7	437.1	429.4	389.9	386.8	415.1	397.1
80	470.7	474.5	475.8	466.8	458.7	411.0	403.4	438.9	430.7
90	498.1	502.4	503.1	499.8	495.8	438.8	424.0	469.1	471.7
FBP	538.2	549.1	550.5	548.0	540.4	508.4	476.2	499.7	518.9

8.0 EVALUATIONS AND BALANCES

Material and heat balances are presented in Tables 6 and 7, in the form specified. Appendix 4 describes the procedure used, as agreed, to obtain these results.

From these tables the heat loss of the unit can be estimated.

Heat Input : 71,067 kJ/h (100%)

Heat Output in Discharge Streams:

Retort Gas and Solids	9,406 kJ/h	(13%)
Combustor Solids	2,272 "	(3%)
Flue Gas Out	27,630 "	(39%)
Cooling water, retort	2,455 "	(4%)
Thus Unit Heat Loss	29,304 "	(41%)

An alternative calculation can be made using heat losses as estimated from Tables 6 and 7. Thus,

Unit Heat Loss = Combustor loss + Retort loss + Heat Exchangers loss

The heat loss for heat exchangers E-2 can be obtained from the tables, but that for heat exchanger E-1 is estimated as 5% of the total input heat. From these data,

The Unit Heat Loss = 26,655 kJ/h, which gives a 10% lower result, hence the higher value is retained.

As noted previously, the small sized UBC unit has a relatively larger surface area for its volume and mass, and this factor should be kept in mind when scale factors are considered. Furthermore, in this experimental apparatus about 40% of the heat input was discharged in the combustor flue gas, and if unit heat losses are reduced this amount could be larger. Perhaps half of this flue gas heat could be recoverable as useful energy.

TABLE 6 - MATERIAL AND HEAT BALANCES - RETORT SYSTEM

Retort	Mass Flow Rate (kg/h)	Temperature (°C)	Unit Enthalpy** (kJ/kg)	Total Stream Enthalpy (kJ/h)
Oil Shale	3.783	20	0	0
Recycle Retort Gas*	26.460	587	635	16,802
Total Entering	30.240			16,802
Net Retort Gas	0.200	98	81	16
Total Retort Exit Gas	26.660	272	282	7,524
Kerogen	0.134	272	738	99
Retort Cyclone Solids	0.006	272	260	2
Spent Shale	3.210	497	550	1,765
Total Leaving	30.010			9,406
IN - OUT	0.220			7,396
Heat Exchanger E-1				
Cooling Water In	1210	13	29	-35,574
Cooling Water Out	1210	13		
Hot Retort Fluids	26.660	136	130	3,464
Cold Retort Fluids	26.660	48	31	836

* - Includes the net nitrogen flow within the retort circuit

** - Since heat of reaction for retorting is zero, enthalpies refer to sensible heat only.

TABLE 7 - MATERIAL AND HEAT BALANCES - COMBUSTOR SYSTEM

Combustor	Mass Flow Rate (kg/h)	Temperature (°C)	Unit Enthalpy (kJ/kg)	Total Stream Enthalpy (kJ/h)
Coal	2.37	20	29,986	71,067
Air	57.70	20	0	0
Spent Shale	2.03	20	0	0
Total Entering	62.10			71,067
Flue Gas	58.60	866	973	57,012
Spent Solids	0.89	911	1054	939
Combustor Cyclone Solids	1.16	866	1149	1,333
Total Leaving	60.60			59,284
IN - OUT	+ 1.44			11,783
Heat Exchanger E-2				
Hot Flue Gas In	58.60	760	851	49,869
Cold Flue Gas Out	58.60	430*	472	27,630
Cold Recycle Retort Gas In	26.46	83	71	1,867
Hot Recycle Retort Gas Out	26.46	587	635	16,803

Enthalpy Datum 20°C

* Estimated

9.0 CONCLUSIONS

A primary objective of the work, that of demonstrating the capability of the Polymath - UBC team and apparatus to perform a 48 hour steady state integrated retorting co-combustion experiment run, was successfully achieved. Some comments follow later in regard to specific points relating to the apparatus.

The oil yield recovery from the retorting system was 44% of the Fischer Assay of the Oil Shale. Considerable carbon remained in the spent shale from the retort overflow, which fed to the combustor as a partial fuel source.

Sulphur capture was also lower than expected, because the CaO in the spent shale was lower than preliminary estimates. Thus the spent shale fed into the combustor gave a Ca/S mol ratio of 0.52, which in turn led to a sulphur capture of about 45%. The terms of the experiment did not include the evaluation of maximum sulphur capture using additional CaO input to the combustor, but it can be expected that increasing the Ca/S mol ratio above unity should lead to a higher capture. Minto coal contains a high sulphur content (approximately 6%).

Minto coal was capable of maintaining combustion. A high volatile coal was found necessary for start-up and run-up to stable operating temperatures.

The test run demonstrated that fluidized bed technology can be applied to the pyrolysis of New Brunswick shale, and that heat from the combustion of Minto coal and spent shale can provide a source of co-generation energy. The low oil yield attained may be accounted for by the presence of large amounts of spent shale in the reactor, promoting cracking or re-deposition of the oil on the spent shale. Future studies could evaluate means of improving oil recovery while improving sulphur capture and heat recovery.

Heat loss from the experimental unit was 29,304 kJ/h. The small size of the unit results in a relatively larger surface area for its volume and mass, leading to a bigger heat loss per unit output. This should be considered in scale-up evaluations.

An evaluation of the heat output shows about 41% is due to the unit heat loss, 13% to the retort gas and solids, 3% to combustor solids, 4% to the retort gas cooling water, and the remaining 39% is discharged in the combustor flue gas. Some part of this flue gas heat loss, perhaps half, can be recoverable as useful energy.

Some additional, if minor points, relative to the apparatus were evident from this work:

- the UBC apparatus arrangement was mainly designed for a direct flow of retorting gas deriving from the combustor below. An indirect arrangement, as for this experiment, loses much of the benefit of the compact design. The unit was successfully modified

but at some effort and more cost than if it had been re-structured to suit the requirements;

- heat exchangers for the gas to gas indirect heating mode had to be the maximum evaluated, in order to obtain the recycled retort gas temperatures essential for the retorting of the shale feed;

- the refractory-lined cyclone in the combustor flue gas stream performed satisfactorily. Heat loss was kept low, and the flue gas entering the gas to gas heat exchanger was maintained at a suitably high level.

On the retort gas outlet stream, a stainless steel cyclone appeared adequate, as long as condensation of oil did not take place in the cyclone. This proved to be the case, though there were other very minor losses of kerogen vapour as described;

- the spent shale discharge feeding into the combustor requires a lock hopper and dedicated feeder to prevent the back flow of combustor gases from affecting the feed rate.

APPENDIX 1

Analyses of Input Material

1a. Proximate and Ultimate Analyses of Coal and Oil Shale

1b. Fischer Assay of Oil Shale Samples

Chemex Labs Ltd.

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• Analytical Chemists • Geochemists • Registered Assayers

Phone: (604) 984-022
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CERTIFICATE OF ASSAY

TO : POLYMATH ENERGY CONSULTANTS LTD. **

718 - 701 W. HASTINGS ST.
VANCOUVER, BC
V6C 1A1

CERT. # : A8712095-001
INVOICE # : I8712095
DATE : 3-APR-87
P.O. # : NONE

Sample ID	BASIS	R.M. %	ASH %	V.M. %	F.C. %	SULPHUR %	CAL. VALUE cal/g
MINTO COAL	AD	1.24	14.68	35.12	48.96	0.31	7162
	DRY	--	14.86	35.56	49.58	0.39	7252
SHALE COMP. 1,2,3,	AD	2.53	78.28	17.75	1.44	0.34	1321
	DRY	--	80.31	18.21	1.48	0.35	1355

ULTIMATE ANALYSIS (*)

Sample ID	BASIS	R.M. %	ASH %	CARBON %	HYDROGEN %	NITROGEN %	SULPHUR %	OXYGEN %
MINTO COAL	AD	1.24	14.68	70.49	4.76	0.98	0.31	2.78
	DRY	--	14.86	71.38	4.68	0.99	0.39	1.70
SHALE COMP. 1,2,3	AD	2.53	78.28	13.11	2.01	0.50	0.34	5.76
	DRY	--	80.31	13.45	1.77	0.51	0.35	3.61

NOTE: *) HYDROGEN AND OXYGEN INCLUDE H AND O IN SAMPLE MOISTURE

V01 M1

W. Kostuik

Registered Assayer, Province of British Columbi

Chemex Labs Ltd.

Analytical Chemists *Geochemists* *Registered Assayers*

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TO: POLYMATH ENERGY CONSULTANTS LTD.
718 - 701 W. HASTINGS ST.
VANCOUVER, BC
V6C 1A1

CERTIFICATE #: A8712095 -002
INVOICE : I8712095
DATE : APRIL 3,1987
P.O. # : NONE

SIZE ANALYSIS:

FRACTION	MINTO COAL	SHALE COMPOSITE 1,2,3
+4 MESH %	5.33	0
4 X 8 MESH %	23.07	33.71
8 X 16 MESH %	27.93	22.59
16 X 30 MESH %	17.57	12.40
30 X 50 MESH %	10.53	7.90
50 X 100 MESH %	7.63	6.34
100 X 200 MESH %	3.27	4.63
200 MESH X 0 %	4.67	12.43

ASH ANALYSIS :

SiO2 %	33.66	64.86
Al2O3 %	10.65	16.24
TiO2 %	0.58	0.75
Fe2O3 %	48.06	6.92
MgO %	0.31	1.97
CaO %	3.62	5.22
Na2O %	0.17	1.70
K2O %	0.77	2.04
P2O5 %	1.97	0.60
SO3 %	1.80	0.92

Certified by W. Stanbridge

Report Number: F/86/294 Date: 1987 - 03 - 26

Job Number: 03001122/0001

Client:

Mr. W. B. Alderton
Polymath Energy Consultants Ltd.
Suite 718
744 West Hastings Street
Vancouver, British Columbia
V6C 1A5

Oil Shale Analysis

Three samples of oil shale were received by Department 3-0, RPC on Monday, March 9, 1987 for a requested Fischer assay.

The shales were assayed using a modified Fischer retort as described in US Department Interior, Bureau of Mines Report of Investigations #3977.

RESULTS:

	Sample #1 -20 mesh +400 mesh	Sample #2 -8 mesh +20 mesh	Sample #3 -4 mesh +8 mesh
Wt. Shale Sample (g)	100.0	100.0	100.0
Total Shale Loss (g/sample)	13.5	15.0	14.5
Wt. Oil Recovered (g)	3.5539	8.0598	9.5280
Density Oil (g/ml @ 15.5°C)	0.8743	0.8740	0.8770
% Oil Yield	3.6	8.1	9.5
% Water Yield	5.8	4.7	4.0
% Gas Lost	4.1	2.2	1.0
Oil Yield (Imp. gal/ton)	8.1	18.4	21.7

C. Bagnell,
Food Technologist

G. D. Brown,
Senior Consultant

/dlp

APPENDIX 2

Analyses of Output Samples Collected during Experiment

2a. Analysis of Ash Samples.
Elemental Carbon and Sulphur in Ash Samples
Size Analysis of Ash Samples

2b. Report on Shale Retort Liquids
Weight Yields, API density, and Ash Content
Gas Analyses for Retort and Combustot Gas Samples
Distillation Results for Shale Oil Samples Collected
Distillation Results for Condenser and Extract Samples

Chemex Labs Ltd.
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PHONE (604) 984-0221

To: POLYMATH ENERGY CONSULTANTS LTD.

718 - 744 W. HASTINGS ST.
VANCOUVER, BC
V6C 1A5

A8719197

Comments:

CERTIFICATE A8719197

POLYMATH ENERGY CONSULTANTS LTD.
PROJECT : SHALE
P.O.# : NONE

Samples submitted to our lab in Vancouver, BC.
This report was printed on 28-AUG-87.

SAMPLE PREPARATION

CHEMEX CODE	NUMBER SAMPLES	DESCRIPTION
214	35	Received sample as pulp

ANALYTICAL PROCEDURES

CHEMEX CODE	NUMBER SAMPLES	DESCRIPTION	METHOD	DETECTION LIMIT	UPPER LIMIT
833	36			0.01	N/A
837	36			0.01	N/A
851	36	SiO2 %: Coal whole rock	ICP-AES	0.01	99.00
852	36	Al2O3 %: Coal whole rock	ICP-AES	0.01	99.00
853	36	Fe2O3 %: Coal whole rock	ICP-AES	0.01	99.00
854	36	MgO %: Coal whole rock	ICP-AES	0.01	99.00
855	36	CaO %: Coal whole rock	ICP-AES	0.01	99.00
856	36	Na2O %: Coal whole rock	ICP-AES	0.01	99.00
857	36	K2O %: Coal whole rock	ICP-AES	0.10	99.00
858	36	TiO2 %: Coal whole rock	ICP-AES	0.001	99.00
859	36	P2O5 %: Coal whole rock	ICP-AES	0.010	99.00
860	36	SO3 %: Coal whole rock	GRAVIMETRIC	0.001	99.00

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To: POLYMATH ENERGY CONSULTANTS LTD.
718 - 744 W. HASTINGS ST.
VANCOUVER, BC
V6C 1A5
Project : SHALE
Comments:

*Page No. : 1
Tot. Pages: 1
Date : 25-AUG-87
Invoice # : I-8719197
P.O. # : NONE

CERTIFICATE OF ANALYSIS A8719197

SAMPLE DESCRIPTION	PREP CODE		ASH I %	S I %	SiO2 %	Al2O3 %	Fe2O3 %	MgO %	CaO %	Na2O %	K2O %	TiO2 %	P2O5 %	SO3 %		
CBM 21/7 00.45	214	—	98.04	0.36	77.00	8.57	6.27	1.09	3.33	0.83	1.20	0.380	0.490	0.890		
CBM 21/7 05.00	214	—	99.77	0.65	62.16	14.88	8.84	1.97	4.57	1.34	2.30	0.700	0.740	1.640		
CBM 21/7 11.00	214	—	99.89	0.64	61.98	15.87	8.44	2.02	4.54	1.43	2.00	0.730	0.700	1.700		
CBM 21/7 17.00	214	—	99.75	0.76	61.31	15.36	8.31	2.01	5.18	1.43	2.30	0.710	0.720	1.960		
CBM 21/7 23.00	214	—	99.75	0.85	61.48	14.97	8.00	1.96	5.28	1.52	2.10	0.680	0.680	2.160		
CBM 22/7 05.00	214	—	99.76	0.84	61.24	15.46	8.49	2.09	5.71	1.68	2.20	0.720	0.680	2.100		
CBM 22/7 11.00	214	—	99.66	0.77	61.45	15.24	8.42	2.08	5.41	1.55	2.30	0.690	0.690	1.930		
CBM 22/7 17.00	214	—	99.66	0.91	60.23	14.73	8.09	2.02	5.82	1.51	2.10	0.670	0.680	2.250		
CBM 22/7 22.00	214	—	98.25	0.72	60.76	14.93	8.33	2.05	5.29	1.53	2.50	0.670	0.690	1.810		
CCS 20/7 23.30	214	—	94.52	0.29	75.37	5.72	11.97	0.52	2.54	0.20	1.40	0.280	0.880	0.580		
CCC 21/7 05.00	214	—	96.78	0.47	57.64	12.59	15.05	1.58	4.64	0.96	2.60	0.580	1.010	1.330		
CCC 21/7 11.00	214	—	93.75	0.62	53.37	13.65	18.11	1.56	4.68	1.02	2.00	0.630	1.060	1.630		
CCC 21/7 17.00	214	—	94.43	0.63	54.33	14.06	17.15	1.66	4.59	1.06	1.80	0.660	1.040	1.690		
CCC 21/7 23.00	214	—	94.16	0.69	55.50	14.43	16.30	1.72	4.54	1.17	2.00	0.690	0.990	1.790		
CCC 22/7 05.00	214	—	96.40	0.67	56.13	14.01	14.15	1.82	5.13	1.22	3.10	0.640	0.990	1.690		
CCC 22/7 11.00	214	—	94.72	0.57	52.90	13.99	19.72	1.50	4.65	0.98	2.00	0.660	1.130	1.480		
CCC 22/7 17.00	214	—	95.28	0.61	55.57	14.26	16.93	1.69	4.46	1.17	1.70	0.680	1.040	1.640		
CCC 22/7 22.00	214	—	94.68	0.57	53.28	13.75	19.84	1.55	4.65	1.08	2.10	0.660	1.140	1.560		
RCS 20/7 23.45	—	—	88.60	0.28	63.39	15.60	6.95	2.19	4.52	1.52	3.00	0.710	0.720	0.650		
RCS 21/7 00.15	214	—	85.56	0.44	64.41	13.52	7.19	1.86	4.72	1.62	2.30	0.620	0.810	1.160		
RCS 21/7 05.00	214	—	84.42	0.37	63.67	14.82	6.83	1.98	4.60	1.74	2.60	0.660	0.720	0.870		
RCS 21/7 11.00	214	—	81.76	0.35	61.63	15.64	7.98	2.05	3.43	1.75	3.00	0.710	0.600	0.860		
RCS 21/7 17.00	214	—	80.07	0.37	59.82	15.88	9.83	2.09	3.40	1.48	3.10	0.710	0.650	0.910		
RCS 21/7 23.00	214	—	78.16	0.41	62.50	16.17	8.60	2.12	3.23	1.58	2.40	0.820	0.620	0.990		
RCS 22/7 05.00	214	—	85.34	0.43	61.53	15.82	8.33	2.06	3.65	1.66	2.60	0.710	0.670	1.130		
RCS 22/7 ———	214	—	80.06	0.44	59.85	16.09	8.60	2.08	3.24	1.50	2.80	0.720	0.610	1.060		
RCS 22/7 17.00	214	—	77.86	0.40	59.60	16.43	10.23	2.08	3.19	1.47	2.20	0.750	0.590	1.020		
RCS 22/7 22.00	214	—	80.36	0.40	58.42	14.49	8.25	1.80	2.99	2.79	2.90	3.290	0.640	1.030		
SS 21/7 05.45	214	—	84.70	0.28	62.33	15.76	6.83	2.15	4.54	1.51	2.20	0.730	0.660	0.600		
SS 21/7 11.00	214	—	83.48	0.29	63.53	15.88	6.42	2.18	4.95	1.68	2.30	0.720	0.620	0.580		
SS 21/7 17.00	214	—	85.05	0.31	62.73	15.28	6.31	2.16	5.54	1.72	2.30	0.690	0.630	0.580		
SS 21/7 23.00	214	—	86.02	0.32	63.23	15.87	6.41	2.19	5.46	1.70	2.20	0.720	0.640	0.640		
SS 22/7 05.00	214	—	84.68	0.29	63.25	15.65	6.45	2.19	5.17	1.72	2.30	0.710	0.630	0.570		
SS 22/7 11.00	214	—	82.55	0.31	63.45	15.85	6.41	2.17	5.16	1.69	2.30	0.720	0.630	0.590		
SS 22/7 17.00	214	—	82.31	0.31	63.59	15.94	6.52	2.23	5.18	1.71	2.50	0.730	0.640	0.580		
SS 22/7 22.00	214	—	81.94	0.32	63.01	15.69	6.49	2.20	5.25	1.68	2.60	0.720	0.660	0.620		

CERTIFICATION : Anna Christie

CHEMEX LABS LTD.

212 BROOKSBANK AV.
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CANADA V7J 2C

TELEPHONE (604) 984 02
TELEX 043 52

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CERTIFICATE OF ANALYSIS

TO: POLYMATH ENERGY CONSULTANTS LTD.
718 - 744 W. HASTINGS ST.
VANCOUVER, BC
V6C 1A5

CERTIFICATE #: A8719197-001
INVOICE #: 18719197
DATE : 24-AUG-87
PROJECT: SHALE

ASH, TOTAL CARBON, TOTAL SULPHUR % as received :

GROUP 1 COMBUSTOR BED MATERIAL

#	Date	Time	grams rec'd	R.M.%	ASH%	Carbon%	Sulphur%
3	21/7	00:45	458	0.06	98.04	1.73	0.355
4		05:00	982	0.06	99.77	0.32	0.654
5		11:00	600	0.06	99.89	0.20	0.641
6		17:00	851	0.06	99.75	0.23	0.765
7		23:00	631	0.06	99.75	0.15	0.847
8	22/7	05:00	588	0.05	99.76	0.23	0.842
9		11:00	603	0.05	99.66	0.32	0.773
10		17:00	715	0.08	99.66	0.28	0.914
11		22:00	549	0.08	98.25	1.44	0.721
GROUP 2 COMBUSTOR CYCLONE SOLIDS							
12	20/7	23:30	558	0.17	94.52	5.23	0.289
14	21/7	05:00	610	0.12	96.78	3.07	0.473
15		11:00	1803	0.16	93.75	5.74	0.618
16		17:00	595	0.19	94.43	5.21	0.628
17		23:00	504	0.19	94.16	5.53	0.689
18	22/7	05:00	866	0.09	96.40	3.48	0.606
19		11:00	1774	0.18	94.72	5.00	0.574
20		17:00	545	0.19	95.28	4.42	0.605
21		22:00	936	0.13	94.68	5.10	0.572
GROUP 3 RETORT CYCLONE SOLIDS							
13	20/7	23:45	228.6	0.57	88.60	7.26	0.381
22	21/7	00:15	40.1	0.81	85.56	9.77	0.437
23		05:00	38.4	0.97	84 42	9.52	0.467
24		11:00	83.3	0.80	81.76	11.60	0.548
25		17:00	147.4	0.95	80.07	12.90	0.365
26		23:00	47.5	1.07	78.16	14.20	0.411
27	22/7	05:00	35.9	0.89	85.34	9.59	0.430
28		?	23.0	1.02	80.06	13.20	0.437
29		17:00	13.2	1.15	77.86	14.90	0.400
30		22:00	2.3	0.90	80.36	13.10	0.402

CTA MEMBER CANADIAN TESTING ASSOCIATION

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CERTIFICATE OF ANALYSIS

TO: POLYMATH ENERGY CONSULTANTS LTD.
718 - 744 W. HASTINGS ST.
VANCOUVER, BC
V6C 1A5

CERTIFICATE #: A8719197-002
INVOICE #: I8719197
DATE : 24-AUG-87
PROJECT: SHALE

A. ASH, TOTAL CARBON, TOTAL SULPHUR % as received:

GROUP 4 SPENT SHALE

#	Date	Time	grams rec'd	R.M.%	ASH%	Carbon%	Sulphur%
31	21/7	05:45	283.1	0.56	84.70	9.74	0.279
32		11:00	1364.6	0.44	83.48	10.60	0.292
33		17:00	818.2	0.44	85.05	8.92	0.306
34		23:00	442.4	0.64	86.02	8.08	0.316
35	22/7	05:00	792.9	0.53	84.68	9.10	0.289
36		11:00	722.1	0.55	82.55	10.60	0.315
37		17:00	1089.3	0.65	82.31	10.70	0.313
38		22:00	482.3	0.80	81.94	11.00	0.323

B. SIZE ANALYSIS % FRACTIONAL RETAINED ON SCREEN

#	+4M	+8	+16	+30	+50	+100	+200	-200
3	0.1	1.2	49.6	27.3	21.4	0.3	0.1	L0.1
4	0.4	1.6	75.4	21.7	0.5	0.1	0.1	0.2
5	--	1.7	76.8	20.1	0.6	0.3	0.2	0.3
6	--	2.0	79.4	17.4	0.6	0.2	0.2	0.2
7	--	2.0	79.5	17.4	0.6	0.2	0.2	0.2
8	--	1.9	76.9	19.7	0.5	0.4	0.4	0.2
9	--	1.9	76.0	20.7	0.5	0.4	0.3	0.2
10	--	2.1	78.3	18.3	0.5	0.3	0.2	0.3
11	--	2.6	78.1	18.1	0.4	0.2	0.3	0.3
12	--	--	4.5	24.9	47.4	2.7	2.6	17.9
13	--	0.1	59.4	38.8	1.2	0.2	0.1	0.2
14	--	0.1	18.4	43.4	8.3	5.3	5.6	18.9
15	--	L0.1	15.8	30.5	7.1	6.5	8.5	31.5
16	--	L0.1	15.8	33.1	9.0	7.7	6.7	27.7
17	--	0.2	22.0	30.5	5.8	5.2	6.5	29.8
18	--	0.1	26.3	35.6	5.9	4.9	4.6	22.6
19	--	L0.1	10.5	30.4	7.1	6.7	7.9	37.3
20	--	L0.1	15.3	34.8	5.0	4.8	6.5	33.5
21	--	--	12.6	26.7	6.0	7.8	9.6	37.3
22	--	--	L0.1	0.5	3.5	11.5	19.2	65.2
23	--	--	--	0.2	6.3	17.5	12.8	63.2
24	--	--	--	0.5	5.0	16.8	9.8	67.9
25	--	--	0.1	0.6	7.1	23.2	29.5	39.5
26	--	--	--	0.8	4.7	12.3	6.6	75.6
27	--	--	--	1.1	0.9	16.2	8.7	72.6

MEMBER
CANADIAN TESTING
ASSOCIATION

CERTIFIED BY .....

CHEMEX LABS LTD.

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TELEX: 043-52597

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CERTIFICATE OF ANALYSIS

TO: POLYMATH ENERGY CONSULTANTS LTD.
718 - 744 W. HASTINGS ST.
VANCOUVER, BC
V6C 1A5

CERTIFICATE #: A8719197-003
INVOICE #: I8719197
DATE : 24-AUG-87
PROJECT : SHALE

3. SIZE ANALYSIS % FRACTIONAL RETAINED ON SCREEN

#	+4M	+8	+16	+30	+50	+100	+200	-200
28	--	--	0.9	1.3	5.2	10.9	6.5	75.2
29	--	--	2.3	1.5	4.6	10.1	9.2	72.3
30	--	--	8.3	4.2	8.3	8.4	25.0	45.8
31	--	0.3	66.4	31.7	1.1	0.1	0.1	0.3
32	--	0.4	66.2	31.2	1.6	0.2	0.1	0.3
33	--	0.3	71.8	26.5	0.9	0.1	0.1	0.3
34	--	0.2	61.9	36.6	0.8	0.1	0.1	0.3
35	--	0.2	61.1	37.2	0.9	0.1	0.2	0.3
36	--	0.4	69.8	29.2	0.3	0.1	0.1	0.1
37	--	0.4	65.6	33.1	0.5	0.1	0.2	0.1
38	--	0.5	70.7	28.1	0.4	0.1	0.1	0.1

NOTE: L DENOTES A LESS THAN VALUE

CERTIFIED BY .....

MEMBER CANADIAN TESTING ASSOCIATION

TEO
RESEARCH LTD.
372 East 37th Avenue, Vancouver, B.C. Canada, V5W 1E7 • Telephone (604) 325-9234

September 26, 1987

Mr. Charles E. Wood, P.E.
President
Polymath Energy Consultants Ltd.
Suite 220, 744 West Hasting Street
Vancouver, B.C. V6C 1A5

Dear Charles,

Re: DSS Oil Shale Retort Project
Revised version of the Gas Analysis Report (20-22 July, 1987)

Enclosed please find the revised data on the combustion flue gas CO2 concentrations. The original values from the data station used a linear calibration line based on 15% CO2, that is where the problem arises. The revised data is based on a calibration curve derives from 1.0, 7.5, 10.0 and 15.0 volume % of CO2 in air.

Please discard the old version and substituted with this revised version. If you have any question direct towards this report please do not hesitate to call me.

Yours truly,
TEO RESEARCH LTD.

K.C. Teo

K.C. Teo, Ph.D.
Managing Director

GAS ANALYSIS REPORT (revised version)

POLYMATH: OIL SHALE RETORT/ DSS PROJECT
Date: 20-22 July, 1987 Revised on 25 Sept., 1987

Retort Gas Sample

Sampling date: July -		20	21	21	21	21
Sampling time: Hour		23:01	05:15	11:00	18:00	22:35
Gas /concentration by volume						
Hydrogen,	ppm	1,463	1,484	338	1,292	1,716
Oxygen,	%	0.59	0.59	1.32	3.18	0.37
Nitrogen,	%	----------		balance		----------
CO_2	%	1.70	2.86	5.37	1.89	2.06
CO	ppm	1,545	2,125	4,446	2,708	1,224
CH_4	ppm	926	849	767	771	729
C_2H_6	ppm	266	259	216	262	761
C_2H_4	ppm	290	205	369	265	285
C_2H_2	ppm	0	0	148	154	139
C_3's	ppm	666	710	667	1,745	1,049
C_4's	ppm	490	246	813	1,364	1,046
H_2S	ppm	14	3	0	0	44
SO_2	ppm	0	0	0	0	0

Combustion Gas Sample

Sampling date: July -		20	21	21	21	21
Sampling time: Hour		23:00	05:20	11:00	18:10	22:30
Gas /concentration by volume						
Oxygen,	%	10.15	12.17	13.26	9.41	9.60
Nitrogen,	%	----------		balance		----------
CO_2	%	**9.50**	**7.20**	**5.80**	**6.82**	**6.90**
CO	ppm	131	97	52	0	180
CH_4	ppm	0	0	0	0	0
SO_2	ppm	1,788	851	1,693	1,384	1,368
NO_x	ppm	125	142	159	135	130

GAS ANALYSIS REPORT (revised version)

POLYMATH: OIL SHALE RETORT/ DSS PROJECT
Date: 20-22 July, 1987 Revised on 25 Sept., 1987

Retort Gas Sample

Sampling date: July -	22	22	22	22	average
Sampling time: Hour	05:02	10:55	15:56	20:05	(std.dev., ±)
Gas /concentration by volume					
Hydrogen, ppm	2,199	1,063	1,280	1,084	1,324 (497)
Oxygen, %	0.61	1.03	1.49	1.37	1.17 (0.81)
Nitrogen, %	----------	----------	balance	----------	----------
CO_2 %	1.39	2.28	0.97	1.23	2.19 (1.25)
CO ppm	1,836	2,160	1,613	0	2,193 (914)
CH_4 ppm	1,182	671	629	680	800 (160)
C_2H_6 ppm	417	157	165	296	311 (175)
C_2H_4 ppm	421	323	269	659	343 (126)
C_2H_2 ppm	0	0	29	287	151 (82)
C_3's ppm	2,527	1,017	1,214	1,631	1,247 (586)
C_4's ppm	1,624	180	610	320	744 (481)
H_2S ppm	0	0	2	2	7.2 (13.7)
SO_2 ppm	6	10	0	0	1.8 (3.5)

Combustion Gas Sample

Sampling date: July -	22	22	22	22	average
Sampling time: Hour	05:15	10:53	16:02	20:10	(std.dev., ±)
Gas /concentration by volume					
Oxygen, %	12.82	12.70	12.00	12.52	11.63 (1.40)
Nitrogen, %	----------	----------	balance	----------	----------
CO_2 %	**5.23**	**6.95**	**5.50**	**8.35**	**6.92** (1.29)
CO ppm	127	208	130	757	187 (210)
CH_4 ppm	0	0	0	0	0
SO_2 ppm	516	1,196	1,430	1,569	1,311 (383)
NO_x ppm	125	160	130	135	138 (13)

GAS ANALYSIS REPORT (revised version)

POLYMATH: OIL SHALE RETORT/ DSS PROJECT
Date: 20-22 July,1987 Revised on 25 Sept., 1987

(1) Gas analysis conditions:

Retort gas and combustion gas samples were analysed over Varian Gas Chromatograph (model 6400/ 1983) equipped with TCD, methanizer and FID detectors.

* Carrier gas: Argon 25 ml/min., at inlet pressure of 80 psi.
* Temperature programming: 40^{0}C (isothermal for 5 min.); heating at 20^{0}C/min to 140^{0}C; holding isothermal at 140^{0}C for 15 min.
* Column used: (a) Porapak N (2 meters x 1/8"); (b) Molecular Sieve 5A (0.5 meter x1/8").

with series and by-pass configuration with respect to (b). Hydrogen and oxygen were analysed under series configuration over column (b) using TCD detector. CO, CO_2 and all C_1 to C_4 hydrocarbon gases were analysed under by-pass mode over the column (a) only. CO and CO_2 after seperation over column (a) were methanized over Ni-catalyst and detected by FID as CH_4.

* SO_2 and H_2S were analysed over Carbosieve-S (2 meters x 1/8") under 50^{0}C isothermal conditions on Varian GC (model 6000/ 1984) equipped with flame photometric detector (FPD).
* NOx was analysed over Nitrogen Oxides Analyser (model 8840/ 1987) by Monitor Lab Inc. on 4-liter of grap samples.
* Commercial calibration standard gases were used for most of the gas species.

(2) There are always possibility to have atmospheric oxygen diffuses to gas sampling syringes. Therefore, one could replace the GC analysed oxygen data with the readings from on-line oxygen meter.

(3) For the start-up heating period, prior to the shale feeding, the flue gas has the following average composition:

Sampling time: 19:45 Hr (20 July, 1987).

Oxygen : 13:05 %; CO_2: 7.74%; CO: 530 ppm;
Methane: 45 ppm; SO_2: 1,829 ppm; NO_x: 155 ppm.

Yours Truly
TEO RESEARCH LTD.

K.C. Teo, Ph.D.

c.c. A.P.W.
C.E. Wood

TEO
RESEARCH LTD.
372 East 37th Avenue, Vancouver, B.C. Canada, V5W 1E7 • Telephone (604) 325-9234

August 10.1987

Mr. Charles E. Wood, P.E.
President
Polymath Energy Consultants Ltd.
Suite 220, 744 West Hasting Street
Vancouver, B.C. V6C 1A5

Dear Mr. Wood:

Enclosed please find a copy of the REPORT ON SHALE RETORT LIQUID (Part 1). This report summarised the weight yield of retort liquids, the API-density and the ash content (ASTM method) for the liquid samples collected.

If you have any question regarding the report, please do not hesitate to contact me at 228-3238/UBC or 325-9234/home office.

As you and Mr. W.B. Alderton have agreed, I need to analyse the eight collected retort liquid samples over the 48-hour test run period. It is the intention of this letter to confirm about this matter.

Yours sincerely
TEO RESEARCH LTD.

K.C. TEO, PH.D.
Managing Director

REPORT ON SHALE RETORT LIQUID (Part 1)

August 10, 1987

POLYMATH: OIL SHALE RETORT /DSS PROJECT

Tar sample label	Sampling date/ Time, HR	Weight, gram (a)	Weight % distribution	Ash content wt.% (b)
No. 1	21 July/ 05:00	561	8.73	4.80
2	21 July/ 11:00	130	2.02	3.37
3	21 July/ 17:00	534	8.31	2.41
4	21 July/ 23:00	449	6.99	3.46
5	22 July/ 05:00	519	8.07	3.51
6	22 July/ 11:00	289	4.50	2.67
7	22 July/ 17:00	264	4.11	1.25
8	22 July/ 22:00	280	4.36	1.81
	Sub-total	3,026 g.	47.08 %	--
Condenser (c)	48-hr. period	2,410	37.49	2.23
Extract W1 (d)	48-hr. period	460	7.16	3.99
Extract W2 (e)	48-hr. period	532	8.28	9.71
	Total	**6,428 g.**	**100.01%**	overall weight average= **3.39%**

(a) Combined weight from the air-blower condensate and demister condensates.

(b) As received basis, by ASTM method D-482.

(c) From the collector of the condenser.

(d) Concentrated extract of tetrahydrofuran (THF) washout from all pipings **down**stream from the condenser.

(e) Concentrated extract of THF washout from all pipings **up**stream from the condenser, including the condenser.

REPORT ON SHALE RETORT LIQUID (Part 1)

August 10, 1987

Tar sample label	Sampling date/ Time, HR (a)	Specific Gravity $60^0F/60^0F$	API Density (b)
No. 1	21 July/ 05:00	0.9372	19.48
2	21 July/ 11:00	0.9360	19.67
3	21 July/ 17:00	0.9306	20.55
4	21 July/ 23:00	0.9377	19.40
5	22 July/ 05:00	0.9376	19.42
6	22 July/ 11:00	0.9295	20.75
7	22 July/ 17:00	0.9249	21.48
8	22 July/ 22:00	0.9234	21.74
Condenser (c)	48-hr. period	0.9138	23.35
overall weight average (d)		0.9246	21.54
Extract W1 (e)	48-hr. period	-(g)-	--
Extract W2 (f)	48-hr. period	-(g)-	--

(a) Combined weight from the air-blower condensate and demister condensates.

(b) API density = 141.5/Sp. Gr. ($60^0F/60^0F$) - 131.5

(c) From the collector of the condenser.

(d) Not including W1 and W2.

(e) Concentrated extract of tetrahydrofuran (THF) washout from all pipings **down**stream from the condenser.

(f) Concentrated extract of THF washout from all pipings **up**stream from the condenser, including the condenser.

(g) Due to contamination with trace amount of THF solvent.

REPORT ON SHALE RETORT LIQUID (Part 1)

TAR YIELD:

According to the information supplied by Sam Low, the total shale feed over the 48-hour period is 180 kg. Therefore the retort liquid yield (i.e. the tar yield) is

(6.428 kg/180 kg) x 100% = 3.57 %, as received basis

Yours truly,
TEO RESEARCH LTD.

K. C. Teo, Ph.D.
Managing Director

c.c. APW
Jim Iim
Sam Low

REPORT ON SHALE RETORT LIQUID (Part 2)

SIMULATION DISTILLATION CURVES AND TABLES FOR SAMPLES NO. 1 TO 8, CONDENSER, AND EXTRACTS W-1 AND W-2

REPORT DATE: AUGUST 14 AND SEPTEMBER 3, 1987

CHART SPEED 0.5 CM/MIN
ATTEN: 128 ZERO: 10% 5 MIN/TICK

SIMULATION DISTILLATION OF SHALE OIL BY G.C.
SAMPLE ORIGIN: COLLECTED BELOW THE CONDENSER
OVER THE 48 HOUR PERIOD

TITLE: SIM.DIST. OF SHALE OIL/POLYMAT 11:46 3 SEPT 1987
CHANNEL NO: 3 SAMPLE: CONDENSATE METHOD: $D-2887ANAL

VARIAN VISTA 401 - APPLE II

SIMULATED DISTILLATION

METHOD READY:

METHOD: D2887A

MODE: ANALYSIS

SIMDIS ANALYSIS REPORT

TITLE: SIM.DIST. OF SHALE OIL/POLYMAT

VISTA 401 METHOD: $D-2887ANAL

SIMDIS METHOD: D2887A

SAMPLE ID: CONDENSATE

COLUMN: SE-30 CAP.COL.(20METERS)

DATE: 11:46 AUG 12 '87

DETECTOR: FID

OPERATOR: DR. K.C. TEO

TEMP PROGRAM: 30C(2')-9C/'-325C

FLOW RATE: 30 ML / MIN

INJ. VOLUME: 1.0 MICROLITERS

ASTM METHOD: D-2887

CALIBRATION TYPE: R.T.

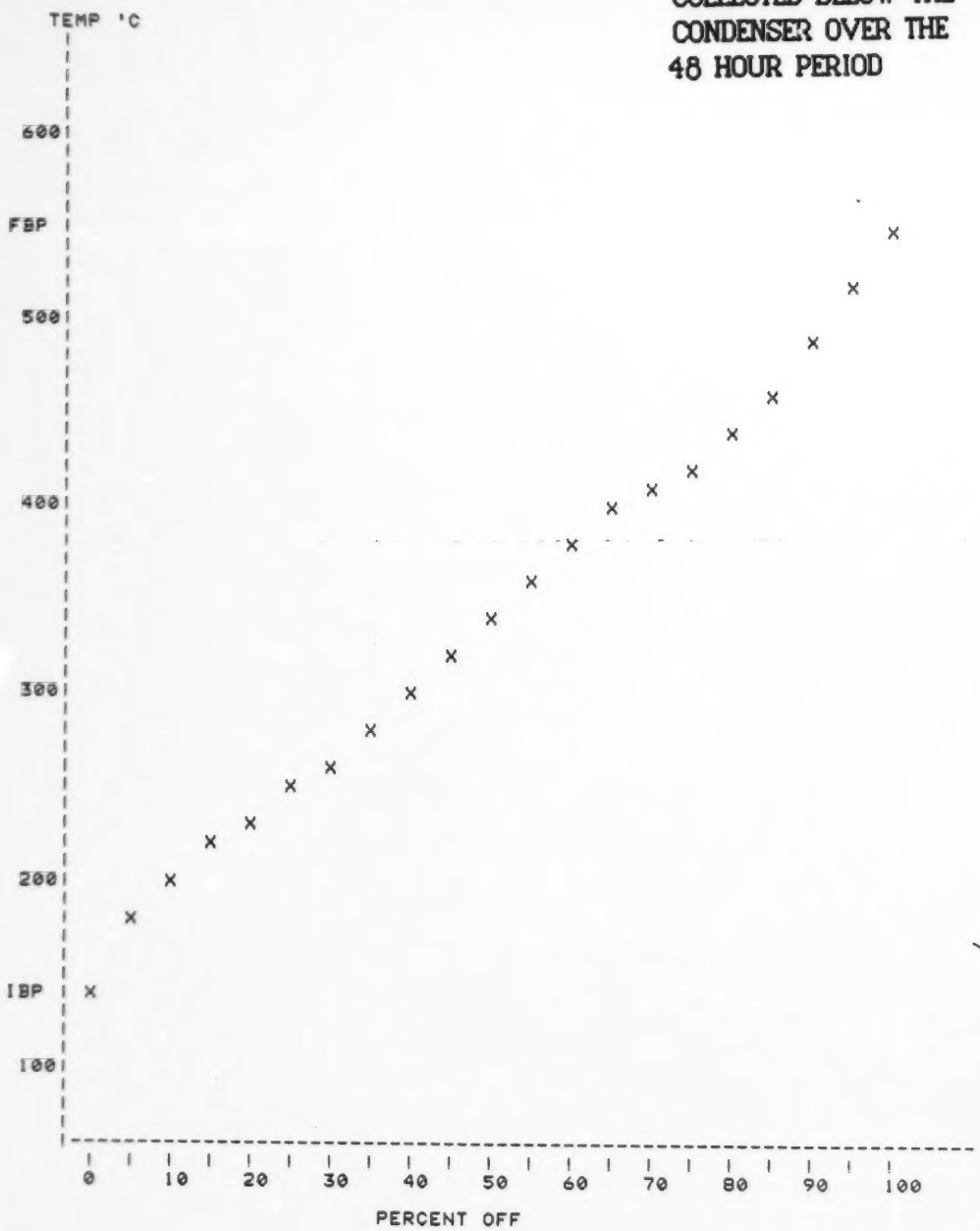
PERCENT OFF	B.P. (DEG. C)
IBP	142
5	185.8
10	206.7
15	221.7
20	236.2
25	252
30	267.9
35	287.6
40	303
45	324.8
50	341.5
55	360.8
60	383.2
65	401.1
70	414.9
75	429.1
80	444.4
85	467.3
90	498
95	525.2
FBP	559.8

D-86 CORRELATION

PERCENT OFF	B.P. (DEG. C)
IBP	216.5
10	243.5
20	263.3
30	285.2
50	338.2
70	397.1
80	430.7
90	471.7
FBP	518.9

ANALYSIS PLOT

SAMPLE ORIGIN:
COLLECTED BELOW THE
CONDENSER OVER THE
48 HOUR PERIOD

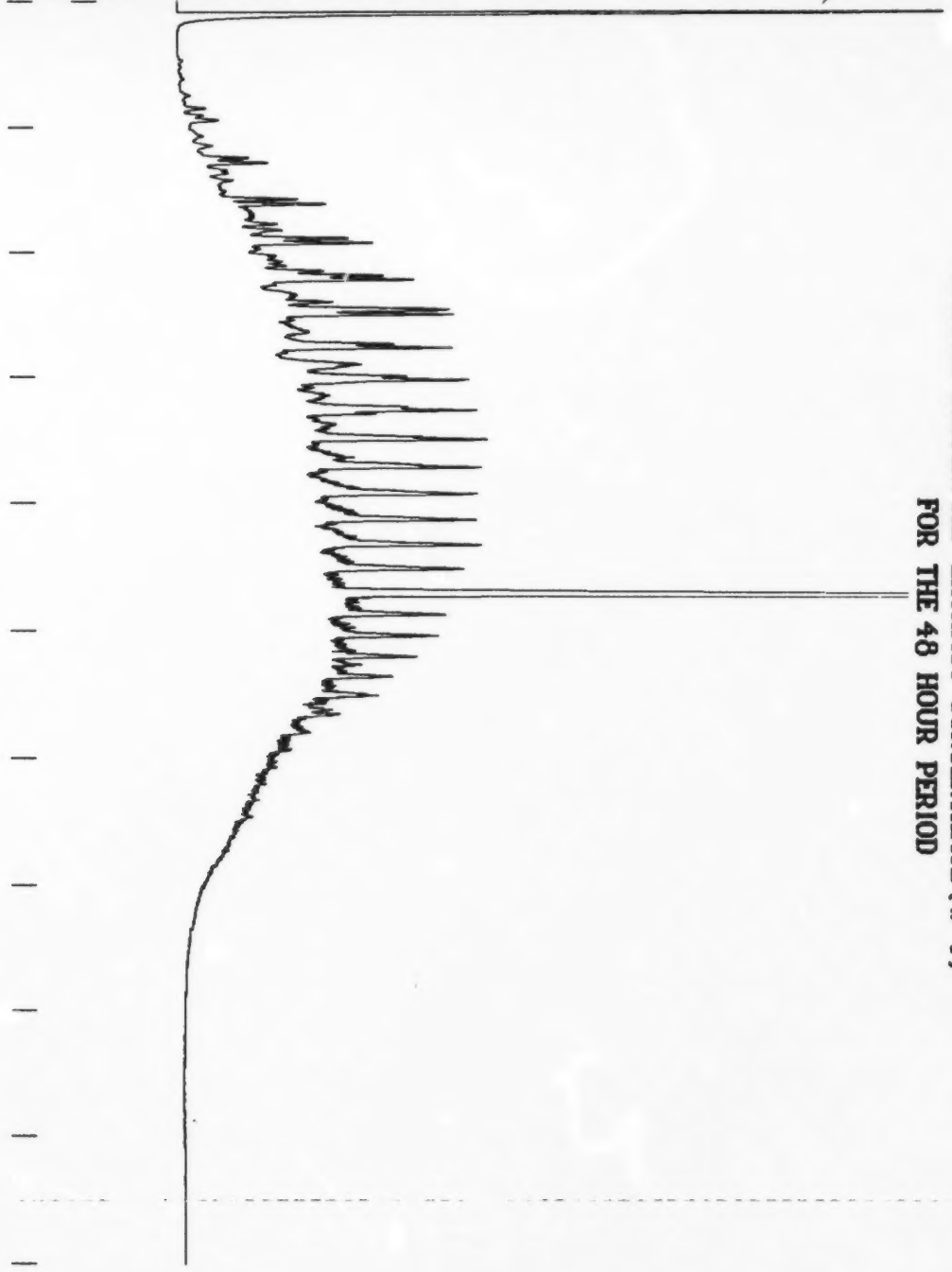


LAST SLICE AREA = -2614
SUM OF AREA = 31677793
SLICE WIDTH = 5 SEC.
END POINT SL#= 2965
ANALYSIS BEYOND THE RANGE OF CALIBRATION

80	7370
85	5239
90	4513
95	19505

CHART SPEED 0.5 CM/MIN
ATTEN: 128 ZERO: 10% 5 MIN/TICK

THF Solvent



SIMULATION DISTILLATION OF SHALE OIL BY G.C.
SAMPLE ORIGIN: THF EXTRACT CONCENTRATE (W-1)
FOR THE 48 HOUR PERIOD

RECALC
TITLE: SIM.DIST. OF SHALE OIL/POLYMAT 1:25 2 SEP 87
CHANNEL NO: 3 SAMPLE: EXTRACT W-1 METHOD: $D-2887ANAL

VARIAN VISTA 401 - APPLE II

SIMULATED DISTILLATION

METHOD READY:

METHOD: D2887A

MODE: ANALYSIS

SIMDIS ANALYSIS REPORT

TITLE: SIM.DIST. OF SHALE OIL/POLYMAT

VISTA 401 METHOD: $D-2887ANAL

SIMDIS METHOD: D2887A

SAMPLE ID: EXTRACT W-1

COLUMN: SE-30 CAP.COL.(20METERS)

DATE: 1:25 SEP 2 '87

DETECTOR: FID

OPERATOR: DR. K.C. TEO

TEMP PROGRAM: 30C(2')-9C/'-325C

FLOW RATE: 30 ML / MIN

INJ. VOLUME: 1.0 MICROLITERS

ASTM METHOD: D-2887

CALIBRATION TYPE: R.T.

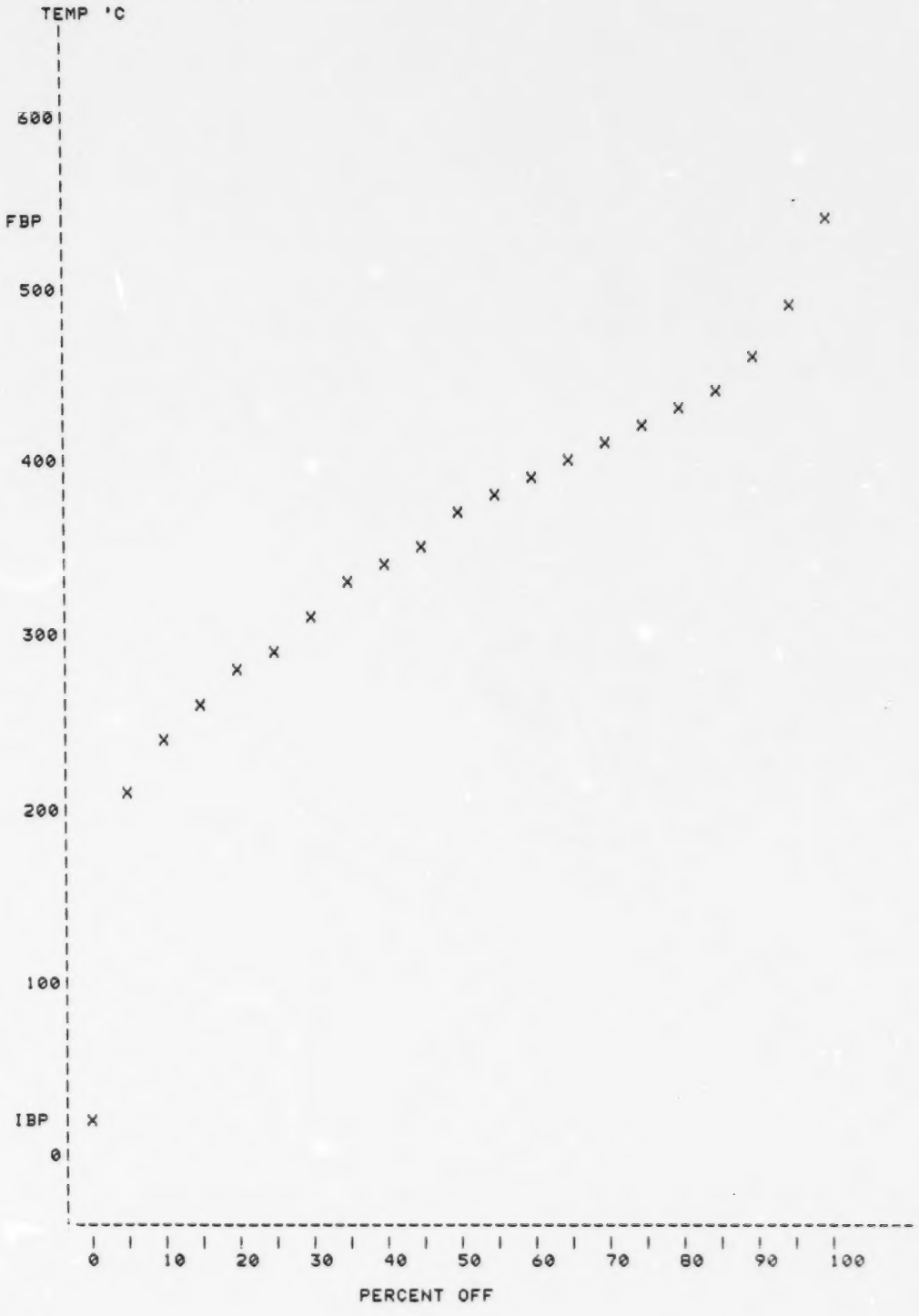
PERCENT OFF	B.P. (DEG. C)
IBP	26.4
5	216
10	242.8
15	263.5
20	281.4
25	297.6
30	312.7
35	330.9
40	341.3
45	355.9
50	370.5
55	384.6
60	397.2
65	404.1
70	410.7
75	421
80	432.2
85	444.1
90	464.7
95	499.5
FBP	542.7

D-86 CORRELATION

PERCENT OFF	B.P. (DEG. C)
IBP	197.3
10	281.6
20	305.5
30	325.8
50	366.5
70	393.9
80	415.8
90	443
FBP	503.8

SAMPLE ORIGIN: THF EXTRACT CONCENTRATE (W-1) FOR THE 48 HOUR PERIOD

ANALYSIS PLOT



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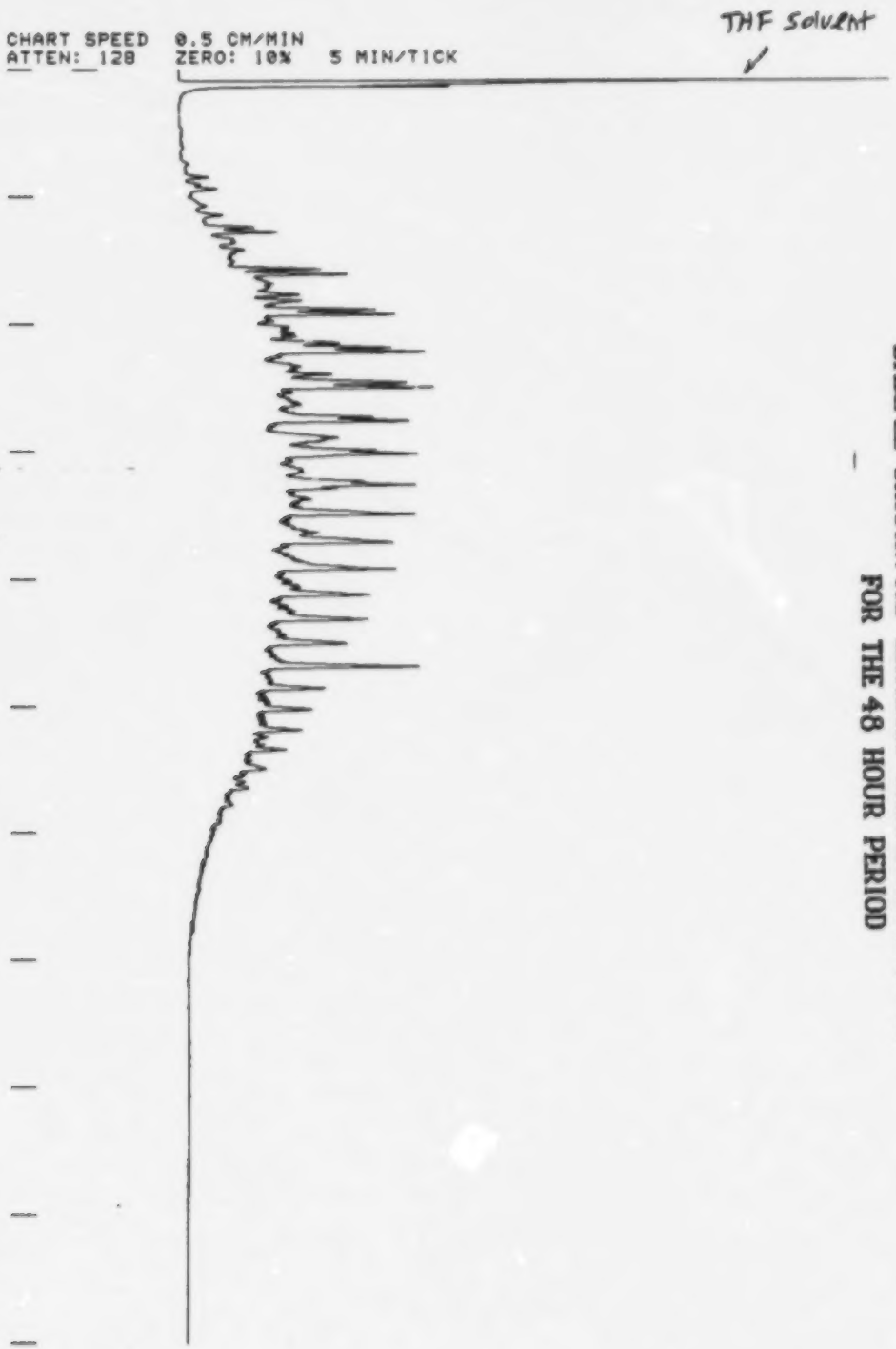
SUM OF AREA = 36292399
SLICE WIDTH = 5 SEC.
END POINT SL#= 2355
ANALYSIS BEYOND THE RANGE OF CALIBRATION



SIMULATION DISTILLATION OF SHALE OIL BY G.C.
SAMPLE ORIGIN: THF EXTRACT CONCENTRATE (W-2)
FOR THE 48 HOUR PERIOD

TITLE: SIM.DIST. OF SHALE OIL/POLYMAT 3:07 2 SEP 87
CHANNEL NO: 3 SAMPLE: EXTRACT W-2 METHOD: @D-2887ANAL

VARIAN VISTA 401 - APPLE II

SIMULATED DISTILLATION

METHOD READY:

METHOD: D2887A

MODE: ANALYSIS

SIMDIS ANALYSIS REPORT

TITLE: SIM.DIST. OF SHALE OIL/POLYMAT

VISTA 401 METHOD: SD-2887ANAL

SIMDIS METHOD: D2887A

SAMPLE ID: EXTRACT W-2

COLUMN: SE-30 CAP.COL.(20METERS)

DATE: 3:07 SEP 2 '87

DETECTOR: FID

OPERATOR: DR. K.C. TEO

TEMP PROGRAM: 30C(2')-9C/'-325C

FLOW RATE: 30 ML / MIN

INJ. VOLUME: 1.0 MICROLITERS

ASTM METHOD: D-2887

CALIBRATION TYPE: R.T.

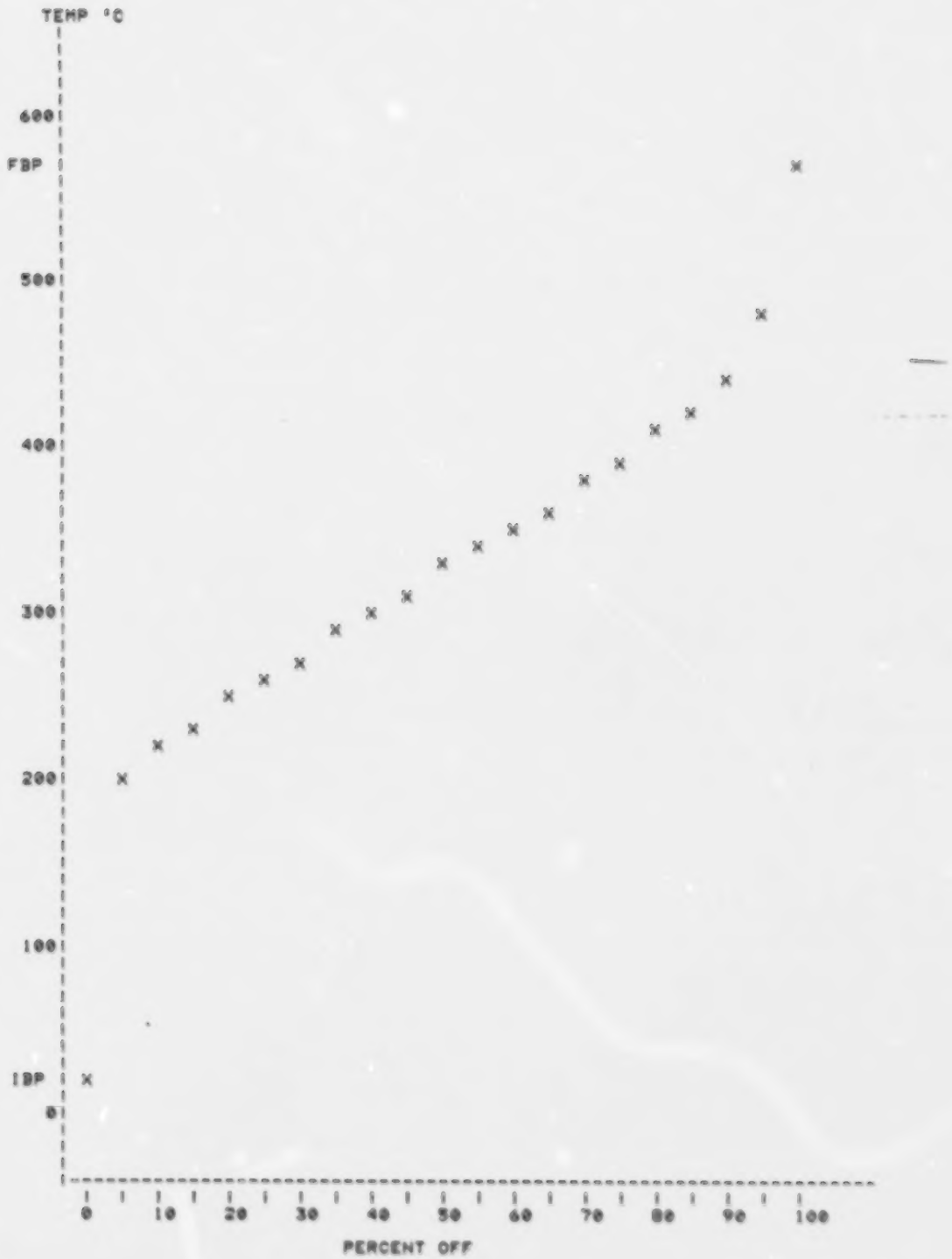
PERCENT OFF	B.P. (DEG. C)
IBP	26.3
5	201.7
10	223.7
15	238.3
20	252.9
25	266
30	278.7
35	292
40	302.4
45	316.3
50	331.9
55	340.9
60	354.3
65	369
70	383.8
75	398.3
80	410.5
85	425
90	441.3
95	482.4
FBP	574.8

D-86 CORRELATION

PERCENT OFF	B.P. (DEG. C)
IBP	186.1
10	259.8
20	276.9
30	292.8
50	328.8
70	370.2
80	391.3
90	423.1
FBP	508.1

SAMPLE ORIGIN: THF EXTRACT CONCENTRATE (W-2) FOR THE 48 HOUR PERIOD

ANALYSIS PLOT



LAST SLICE AREA = 5641
SUM OF AREA = 24508450
SLICE WIDTH = 5 SEC.
END POINT SL#= 2615
ANALYSIS BEYOND THE RANGE OF CALIBRATION

REPORT ON SHALE RETORT LIQUID (Part 3)

September 3, 1987

POLYMATH OIL SHALE RETORT /DSS PROJECT

ELEMENTAL ANALYSIS

Sample label	Sampling date/ Time, HR (a)	Weight % Composition (f) C	H	N	S	O
No. 1	21 July/ 05:00	86.54	11.68	0.99	0.62	0.17
2	21 July/ 11:00	85.87	11.73	0.91	0.77	0.72
3	21 July/ 17:00	85.55	11.58	0.91	0.70	1.26
4	21 July/ 23:00	86.36	11.90	0.98	0.61	0.15
5	22 July/ 05:00	85.00	11.43	0.97	0.86	1.74
6	22 July/ 11:00	85.79	11.64	0.89	0.51	1.17
7	22 July/ 17:00	86.32	11.85	0.83	0.50	0.50
8	22 July/ 22:00	86.15	11.82	0.78	0.50	0.75
Condenser 48-hr. period (b)		86.20	11.59	0.84	0.54	0.83
Overall weight average (c)		**86.05**	**11.64**	**0.89**	**0.60**	**0.82**
Extract W1 48-hr. period (d)		84.65	11.41	0.85	0.57	2.52
Extract W2 48-hr. period (e)		84.83	10.95	0.93	0.64	2.65

(a) Combined weight from the air-blower condensate and demister condensates.

(b) From the collector of the condenser.

(c) Not including W-1 and W-2.

(d) Concentrated extract of tetrahydrofuran (THF) washout from all pipings downstream from the condenser.

(e) Concentrated extract of THF washout from all pipings upstream from the condenser, including the condenser.

(f) Ash-free and dry basis; results for W1 and W2 should treat with caution due to contamination of small amount THF solvent.

(g) Oxygen, by difference =100 - (C+H+N+S) , wt %.

Some notes and comments regarding the elemental analysis:

1. All the C, H and N analyses were performance by Canadian Microanalytical Service Ltd, 1-609 4th Ave., New Westminster, B.C.

2. Sulphur analysis by LECO Sulfur Determinator (model SC-32), calibrated with certified standard oil sample containing 0.89 ± 0.03 wt. % S.

3 The atomic ratio for **C/H =1.61** , excluding samples W-1 and W-2.

Yours truly,
TEO RESEARCH LTD.

K.C. Teo, Ph.D.
Managing Director

c.c. A.P.Watkinson.

APPENDIX 3

Itemized Procedures

3a. Combustor start-up, operating, and shut-down procedures.

3b. Retort start-up, operating, and shut-down procedures.

3c. Emergency procedures.

COMBUSTOR START-UP, OPERATING, AND SHUT DOWN PROCEDURE

1. Turn on propane supply to combustor. The propane tank is located at the corner of the Coal Building, UBC.

2. Turn on the pilot flame air to rotameter setting of 6 and crack open the pilot flame propane valve.

3. Turn on the main air to a rotameter setting of 25.

4. Adjust the reactor pressure to 2 psig by adjusting the gate valve which controls the combustor hot flue gas discharge.

5. The propane gas pressure has to be below 9 psig in order for the propane igniting system to work. The propane pressure can be controlled to a degree by opening the main propane valve slowly while keeping an eye on the propane pressure gauge. A good pressure to have is at about 6 - 7 psig. Turn on the flame detector cooling air valve to a rotameter setting of about 6.

6. Turn on the main electrical power. Reset the ignition controller by pressing the reset button located behind the main panel door. Turn on the ignition switch and wait. After about a minute the white light at the main panel should illuminate to show that the pilot propane solenoid valve is open and propane is being mixed with air. The mixture is then ignited by the spark plug. When the pilot flame is burning the red light should illuminate, and the main propane valve can then be turned on. Turn on the main propane valve to feed propane into the bottom of the combustor.

7. If the pilot flame is <u>not</u> ignited there will be a sharp alarm sounded. Turn off the pilot light ignition switch and wait for about a minute for the gas to clear. Reset the ignition button and repeat step 6.

8. Open the main propane valve to give a rotameter setting of about 6. Let the propane heat the combustor for about 10 to 20 minutes or until the TC#5, (Thermocouple #5 located in the combustor), is about 550^{o}C before introducing 600 grams of pure sand into the combustor slowly, (lower feed setting at 1).

9. The flame front must be lowered to the distributor grid by decreasing the main air flow, and by keeping the combustor pressure at about 2 psig by closing the exit flue gas valve. The sand bed may slug and the combustor pressure may oscillate. Try and keep the variation between 2 to 4 psig to prevent the pilot flame being blown out. The oscillation helps to move the sand bed and helps heat it to higher temperatures.

10. During the heating up period mix up an amount of sand and Forestburg coal,in the proportion of 3.4 kg of sand and 0.68 kg of coal.

11. When TC#4 reads about 400°C the coal/sand mixture is fed slowly to the combustor, (feed setting about 3 - 6), to build up the bed. Make sure the combustor pressure is above 2 psig. Try to keep a slugging bed going to bring the hot surface bed material down to the distributor grid.

12. After the coal/sand mixture is fed into the combustor, feed in pure Forestburg coal to bring the combustor to operating temperature. This will require about 10 kg of Forestburg coal with a feeder setting of 8 - 10. Switch over to the Minto coal when the temperature is about 800°C. Shut off the main propane flow but retain the pilot on in case of reactor cooling during the coal switchover.

13. The recommended Minto feed rate is about setting 9 on the feeder. The air flow rate is adjusted according to the temperature. Try and keep the combustor temperature below 1000°C to prevent clinkering. Turn on the combustor coil cooling to prevent the coil burning through. The valve for this is located behind the combustor near the wall.

14. Start the retort recycle blower to circulate air through the heat exchangers and to preheat the retort section as soon as the combustor temperature is fairly steady. This will usually be about 3 hours after start up. Set the recycle gas flow at setting 3 on the rotameter.

15. Maintain the combustor temperature at 900 -950°C on TC#4,5 and 6, by making adjustments to the combustion air flow rate and coil feeder as necessary. Note any changes made to these settings.

16. Final shut-down of the unit is by stopping the coal feeder while maintaining the air flow. This will cool the bed to a safe shut down temperature in about 15 minutes. Once the bed is at or below 600 - 650°C the air flow may be stopped. If possible the air should be maintained a little longer to give the bed and combustor a lower temperature from which to cool overnight.

RETORT START-UP, OPERATING, AND SHUT-DOWN PROCEDURE

1. When the gas recycle has raised the retort temperature to about 300°C, feed about 5 kg of oil shale slowly to the retort to build up the retort bed. After this shale is fed, stop the feeder.

2. Allow the retort bed temperature to rise until steady at about 500°C, then start a continuous shale feed through the rotary valve. A feeder set point of 4.0 will give the required shale feed rate of 3.5 kg/h.

3. Once continuous shale feed has commenced, open the spent shale overflow valve to the spent shale receiver.

4. Set the recycle gas rotameter to 30 by adjusting the recycle loop valves on the blower. Keep the blower temperature below 100°C and the pressure below 5 psig.

5. Ensure that both demisters are open and that the valves to their drainpots are open to receive any drainage.

6. Keep an eye on the bed differential manometer. It should oscillate slightly. If it is still it indicates inadequate fluidization. Every 30 minutes open the recycle gas valve for a few seconds to "burp" the bed, thus inducing greater fluidization.

7. About every 90 minutes close the spent shale overflow valve, and unscrew the spent shale receiver. Reattach an empty receiver, then reopen the overflow valve. Drain and weigh the spent shale and put it aside for blending with fresh coal. The receiver can be readied for reuse.

8. To shut-down the retort, stop the shale feeder and the combustor coal feeder. Maintain the combustion air flow and the gas recycle flow. After about 15 minutes with no fuel the combustor and retort temperatures will have dropped sufficiently, and the air flow may then be stopped. If possible the air flow can be maintained a little longer to cool the retort further, making it easier for the retort to cool overnight.

EMERGENCY PROCEDURES

1. **Coal feed stops - feed line empty**

   a) Check that there is coal in the feed hopper.
   b) Check that the feeder is operating.
   c) Rap the coal feed line and feeder end plate with a rubber mallet. Observe if coal starts falling through the Pyrex elbow. DO NOT STRIKE THE PYREX GLASS ELBOW.

2. **Coal feed stops - feed line full**

   a) Stop the coal feeder.
   b) Rap the inclined feed line that leads into the combustor, in case the hold-up is here.
   c) If (b) does not work, there is a blockage in the line, possibly due to a sinter in the combustor. Proceed with the following:
      - reduce combustion air flow to a minimal value to reduce combustor cooling.
      - disconnect the Pyrex elbow.
      - try to blow the line clear by increasing the combustor air flow momentarilly. If the coal is packed too tightly to be easily dislodged, try pushing a metal rod or tube down the feed line to prod out or clear the line. Use one or both of these methods to get the line cleared. Make sure any clinker or caked coal is broken away from the inlet port in the combustor.
   d) Time is of the essence, but if the combustor has cooled excessively, (to 600 - 650^{o}C), pour about 500 g of Forestburg coal down the feed line before reconnecting the Pyrex elbow. Restart the coal feeder as soon as the line is reattached and bring the bed back to operating temperature.

3. **Shale feed stops - feed line empty**

   a) Check that there is shale in the feed hopper.
   b) Check that the feeder is operating.
   c) Rap the shale feeder line and feeder end plate with a rubber mallet. Observe if shale starts falling through the Pyrex elbow.

4. **Shale feed stops - feed line full**

   a) Stop the shale feeder.
   b) Rap the inclined shale feed line leading into the retort.
   c) Check that the spent shale overflow valve is open, and that the spent shale hopper is not full.
   d) If (c) appears satisfactory, momentarilly increase the gas flow rate to "burp" the bed and reestablish fluidization.
   e) Repeat steps (b) and (d) until the shale feed recommences.

APPENDIX 4

Mass and Heat Balance Methods

APPENDIX 4

MASS AND HEAT BALANCES

m_{xy} = mass flow rate in kg/h at xy location

a) **Retort Mass Balance**

$$m_S + m_{RRG} = m_{ROS} + m_{RCS} + m_O + m_{RTG} + m_{RL} \quad . . (1)$$

Shale	+	Retort recycle gas	=	Retort overflow solids	+	Retort cyclone solids	+	Retort oil	+	Retort total gas	+	Retort loss

m_S, m_{RRG}, m_{ROS}, m_{RCS}, m_O were measured directly.

$$m_{RTG} = m_{RNG} + m_{RRG} \quad . . . . . .(2)$$

Retort total gas	=	Retort net gas	+	Retort recycle gas

m_{RNG} and m_{RRG} were measured directly.

m_{RL} was determined from the precipitator catch.

b) **Retort Heat Balance**

$$m_S.H_S + m_{RRG}.H_{RRG} = m_{ROS}.H_{ROS} + m_{RCS}.H_{RCS} + m_O.H_O + m_{RTG}.H_{RTG} + Q_{RL} \quad . . . . . .(3)$$

where H = Total Enthalpy = $C_p\,(T-T_{ref}) + H_{c,Tref}$

C_p = Mean Specific Heat; $H_{c,Tref}$ = Heat of Combustion

H_S, H_{ROS}, H_{RCS} were calculated from solid analyses.

H_O was calculated from the elemental oil analysis.

H_{RRG}, H_{RTG} were calculated from gas chromatography results.

Q_{RL} = Heat Loss from shell of the Retort

Q_{RL} was estimated from shell temperature measurements.

c) **Combustor Mass Balance**

$$m_{ROS} + m_C + m_A = m_{COS} + m_{CCS} + m_{FG} + m_{CL} \quad \ldots . (4)$$

Retort Overflow Solids	+ Coal + Air	=	Comb. Ov'flow Solids	+ Comb. Cyclone Solids	+ Flue Gas	+ Combustor loss

m_{ROS}, m_C, m_A, m_{COS}, m_{CCS} were measured directly.

m_{FG} was measured indirectly.

m_{CL} was calculated by difference, and measured if needed.

d) **Combustor Heat Balance**

$$m_{ROS}.H_{ROS} + m_C.H_C + m_A.h_A = m_{COS}.H_{COS} + m_{CCS}.H_{CCS} + m_{FG}.h_{FG} + m_{CL}.H_{CL} + Q_{CL} \quad \ldots . (5)$$

H_{ROS}, H_C, H_{COS}, H_{CCS}, H_{CL} are calculated from compositions of the solids.

h_{FG}, h_A are calculated from gas chromatography analysis, and from Handbook data respectively.

$$h_i = C_{pi} (T - T_{ref})$$

Q_{CL} is determined from shell temperature measurements and literature equations.

e) **Overall Mass and Heat Balances**

Equations (1) and (4) can be combined into an overall mass balance.

The overall heat balance is determined once the balances are made over the three heat exchangers in the system. These balances are straightforward and are not formulated here.

The balances refer to steady state operation. Accumulation of solids in the heat exchangers, reactors, and other piping are estimated as accurately as possible, and the results incorporated in the above mass balances.

Table A, attached hereto, gives the measurement method, location of measurement point, and the analysis used. Refer to Figure 1 for the arrangement of the apparatus.

MASS FLOW RATES MEASUREMENT

App.4

Item	Measurement Method	Location	Analysis
Retort Inputs			
m_S	Rotary valve, checked by hopper weight	(1)	C/P/FA/PRA*
m_{RRG}	Orifice plate	(2)	GC
Retort Outputs			
m_{ROS}	Intermittent solid sampling and weighing	(3)	P/C
m_{RCS}	" " " " "	(4)	P/C
m_O	Intermittent weighing accumulator, demisters	(5,6,7)	UA
m_{RTG}	Calculated by Equation (2).		
m_{RL}	Precipitator catch weights	(7)	P/C
m_{RNG}	Orifice plate	(8)	GC
Combustor inputs			
m_{ROS}	Intermittent solids sampling and weighing	(3)	P
m_C	Rotary valve, checked by hopper weight	(9)	P/UA/PRA
m_A	Rotameter	(10)	
Combustor Outputs			
m_{COS}	Intermittent weighing of accumulator	(11)	P
m_{CCS}	" " " "	(12)	P
m_{FG}	Calculated by N_2 and H balance		GC
m_{CL}	Gas sampling through filter	(14)	DL

Analysis symbols :
P = plasma analysis for ash constituents
C = carbon analysis
FA = Fischer assay
UA = Ultimate analysis
PRA = Proximate analysis
GC = Gas chromatography
DL = Dust loading

HEAT CAPACITY EQUATIONS FOR ENTHALPY BALANCES

Raw Shale

$$C_p = 0.9190 + 7.2936 \times 10^{(-4)} \times T\ (^{o}C) \qquad kJ/kg.K$$

Retort Spent Shale

$$C_p = 0.8761 + 5.5678 \times 10^{(-4)} \times T\ (^{o}C) \qquad "$$

Combustor Overflow Shale Ash

$$C_p = 0.08332 + 3.842 \times 10^{(-4)} \times T\ (^{o}C) \qquad "$$

Gaseous Constituents

N_2	$C_p = 0.9689 + 0.0001922\ (T\ (^{o}C) + 273)$	"
O_2	$C_p = 0.8302 + 0.0002861\ (T\ (^{o}C) + 273)$	"
CO_2	$C_p = 0.6606 + 0.00030\ (T\ (^{o}C) + 273)$	"
$H_2O(v)$	$C_p = 1.6756 + 0.000593\ (T\ (^{o}C) + 273)$	"

Liquid Water

$$C_p = 4.2 \qquad "$$

Shale Oil

$$C_p = 2.93 \qquad "$$

Flue Gas

$$C_p = 1.15 \qquad "$$

Retort Gas

$$C_p = 1.12 \qquad kJ/kg.K$$

APPENDIX 5

Analytical Methods Used

Oil Shale

Fischer Assay using a modified retort as described in US Department of the Interior, Bureau of Mines Report of Investigations #3977.

Proximate Analysis

CV with Benzoic Acid

Ultimate Analysis

Particle Size Analysis by Sieve of Composite Sample

Mineral Analysis of Ash by ICP-AES (Plasma Spectroscopy)

Minto Coal

Proximate Analysis

CV

Ultimate Analysis

Particle Size Analysis by Sieve

Mineral Analysis of Ash by ICP-AES (Plasma Spectroscopy)

Combustor Bed Material, Combustor Cyclone Solids, Retort Cyclone Solids, and Spent Shale

10 Element Ash Analysis by ICP-AES (Plasma Spectroscopy)

Elemental Carbon by Leco Induction Furnace Method

Particle Analysis by Sieve

Shale Retort Liquids

Weight Yield

Ash Content by ASTM method D-482

Specific Gravity and API Density

Simulation Distillation Results by ASTM D-2887 and D-86 methods

C, H, N, Elemental Analysis

S Analysis by Leco Sulfur Determinator

O by difference

GAS ANALYSIS REPORT

POLYMATH: OIL SHALE RETORT
DSS Project

Date: 20-22 July, 1987

(1) Gas analysis conditions:

Retort gas and combustion gas samples were analysed over Varian Gas Chromatograph (model 6400/ 1983) equipped with TCD, methanizer and FID detectors.

* Carrier gas: Argon 25 ml/min., at inlet pressure of 80 psi.
* Teperature programming: 40°C (isothermal for 5 min.); heating at 20°C/min to 140°C; holding isothermal at 140°C for 15 min.
* Column used: (a) Porapak N (2 meters x 1/8");
(b) Molecular Sieve 5A (0.12 meter x1/8").

with series and by-pass configuration with respect to (b). Hydrogen and oxygen were analysed under series configuration over column (b) using TCD detector. CO, CO_2 and all C_1 to C_4 hydrocarbon gases were analysed under by-pass mode over the column (a) only. CO and CO_2 after seperation over column (a) were methanized over Ni-catalyst and detected by FID as CH_4.

* SO_2 and H_2S were analysed over Carbosieve-S (2 meters x 1/8") under 50°C isothermal conditions on Varian GC (model 6000/ 1984) equipped with flame photometric detector (FPD).
* NOx was analysed over Nitrogen Oxides Analyser (model 8840/ 1987) by Monitor Lab Inc. on 4-liter of grap samples.
* Commercial calibration standard gases were used for most of the gas species.

(2) There are always possibility to have atmospheric oxygen diffuses to gas sampling syringes. Therefore, one could replace the GC analysed oxygen data with the readings from on-line oxygen meter.

(3) For the start-up heating period, prior to the shale feeding, the flue gas has the following average composition:

Sampling time: 19:45 Hr (20 July, 1987).

Oxygen: 13:05 %; CO_2: 11.46%; CO: 530 ppm;
Methane: 45 ppm; SO_2: 1,829 ppm; NO_x: 155 ppm.

Yours Truly
TEO RESEARCH LTD.

K.C. Teo

K.C. Teo, Ph.D.

c.c. A.P.W.
S.Low

Some notes and comments regarding the elemental analysis:

1. All the C, H and N analyses were performance by Canadian Microanalytical Service Ltd, 1-609 4th Ave., New Westminster, B.C.

2. Sulphur analysis by LECO Sulfur Determinator (model SC-32), calibrated with certified standard oil sample containing 0.89 $\pm$ 0.03 wt. % S.

3 The atomic ratio for **C/H =1.61** , excluding samples W-1 and W-2.

Yours truly,
TEO RESEARCH LTD.

K.C. Teo, Ph.D.
Managing Director

c.c. A.P.Watkinson.